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EFFECTS OF VAPORIZED DECONTAMINATION SYSTEMS ON SELECTED BUILDING INTERIOR MATERIALS: CHLORINE DIOXIDE

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14. ABSTRACT:
The National Homeland Security Research Center (NHSRC) of the EPA established an Interagency Agreement with the U.S. Army Edgewood Chemical Biological Center (ECBC) to take advantage of ECBC's extensive expertise and specialized research facilities for the decontamination of surfaces contaminated with chemical and biological (CB) warfare agents. The National Homeland Security Research Center formed a collaboration with ECBC in a mutual leveraging of resources, expanding upon ECBC's on-going programs in CB decontamination to more completely address the parameters of particular concern for decontamination of indoor surfaces in buildings following a terrorist attack.

Vaporized hydrogen peroxide (VHP) and chlorine dioxide (ClO_2) have been used to decontaminate indoor surfaces contaminated with anthax and show potential for use in decontaminating indoor surfaces contaminated by chemical agents. This program is specifically focused on the decontamination of the building environment for purposes of restoring a public building to a usable state after a terrorist chemical warfare attack. As building interiors typically contain large surfaces composed of concrete cinder block, wood, steel, carpet, suspension ceiling tile, and painted wallboard, the effort was designed to determine how building materials are affected by the decontaminants. The focus of this technical report is the evaluation of the building interior materials and the fumigant ClO_2. The work discussed in this report was conducted from November 2003 to October 2006, under EPA IAG DW 939917-01-0.

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PREFACE

The work was completed under EPA IAG DW 939917-01-0. The work discussed in this report was started in November 2003 and completed in October 2006.

This report was published though the Technical Releases Office; however, it was edited and prepared in the Decontamination Sciences Branch, Research and Technology Directorate, ECBC.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. Manufacturer names and model numbers are provided for completeness. This technical report may not be cited for purposes of advertisement.

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A program cannot be successfully completed without the contributions of a good team of people. The authors thank the following individuals for their hard work and assistance with the execution of this technical program:

- David Sorrick (ECBC) for his assistance in acquiring the test equipment and the design and construction of the circuit breaker test stations.

- Dr. David Cullinan (SAIC) for preparing many coupon run baskets, coupon measurements, and chain-of-custody forms during the time his assigned laboratory was closed.

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EFFECTS OF VAPORIZED DECONTAMINATION SYSTEMS ON SELECTED BUILDING INTERIOR MATERIALS: CHLORINE DIOXIDE

1. BACKGROUND

The Material Compatibility effort was designed to determine how the decontaminant vapors impact building materials within an enclosed building interior space. Because building interiors contain large surfaces composed of complex material compositions and electrical components, such as circuit breakers, data are needed to determine how such materials are affected by exposure to the decontaminant vapor. Vaporized hydrogen peroxide (VHP) and chlorine dioxide (ClO_2) were selected because these decontamination technologies have been used to decontaminate indoor surfaces contaminated by anthax and/or the technologies show potential for use in decontaminating indoor surfaces contaminated by chemical agents. The representative building interior materials tested were unpainted concrete cinder block, standard stud lumber (wood 2 x 4 in., fir), latex-painted ½ in. gypsum wallboard, ceiling suspension tile, painted structural steel, and carpet. The physical properties of the building materials were measured using ASTM test methods. The Material Compatibility studies also investigated electrical breakers using Underwriters Laboratories (UL) test methods and the breaker components aluminum, copper, and steel. The samples were studied using specialized chemical testing to determine the type of chlorine-containing salts on the metal surface. In addition, visual appearance was documented. This report contains the results for the ClO_2-exposed coupon Material Compatibility tests. The VHP results are documented in a separate report.

2. SUMMARY OF CONCLUSIONS

The bulleted listing contains the summary of conclusions from this technical report. In general, the ClO_2-exposed building materials showed no change in appearance and some minor change in integrity compared to non-exposed samples. The samples were evaluated for outliers using the Dixon's Q-Test in accordance with ASTM Method E 178, and for statistically demonstrated differences using the Welch's *t* test.

- Visual Inspection: No differences were observed for any of six main material type coupons after ClO_2 exposure and aging compared to before ClO_2 exposure.
- Painted Structural Steel: The ClO_2 fumigated structural steel coupons showed no change in the maximum load required to break the samples compared to the controls. Minor differences in tensile strength were reported; however, these differences were due to differences in the cross-section area of the coupons that were within the tolerance limits set for quality control. All samples were above the specified tensile strength requirements of the ASTM test (20% or more). There was no obvious change in the potential for failure of the steel after a fumigation using ClO_2.
- Gypsum Wallboard: No statistically significant difference in the resistance to penetration by a nail was observed between the control and ClO_2 fumigated coupons. The differences noted were well within the 15% variation indicated in the ASTM specification and the standard deviations of the test.
- Ceiling Tile: Exposure to ClO_2 had no statistically significant effect on the force required to break the ceiling tile coupons compared to the controls.
- Carpet: A minor increase for the average tuft bind results with exposure to ClO_2 was apparent, but the difference was smaller than the standard deviations of the individual test results.

- Concrete Cinder Block: The fumigated concrete cinder blocks did not exhibit any changes from the control samples. There was no evidence to indicate that fumigation with ClO_2 had any effect on the cinder blocks.
- Wood: Exposure to high concentrations of ClO_2 for short durations appeared to reduce the tensile strength of the furring strips, causing them to fail more rapidly (2% decrease at half-target, 15% decrease at full-target concentration) and at lower applied forces (+1.4% at half-target, -17% at full-target concentration).
- Circuit Breakers: Exposure to ClO_2 presented a conflicting picture of the effects on circuit breakers. Under the 60 amp challenge, exposed circuit breakers tripped faster than the controls. Under the 30 amp challenge, the circuit breakers tripped slower than the controls. Either situation could present a problem to the user. Failure criteria must be established to determine if this is an acceptable response.
- Residual Analysis on Metals: ClO_2 decomposed on aluminum to yield chloride, chlorite, chlorate, and perchlorate anions. The products from the reaction of ClO_2 with copper and steel surfaces were chloride, chlorate, and perchlorate. The reaction of ClO_2 on steel was the most severe with chloride the most abundant product. The metal chloride, a decomposition product of the other anions, was the most abundant species on each metal. The amount of chloride anions on each metal was similar in the 6 h (1000 ppm) and 12 h (2000 ppm) tests.

3. INTRODUCTION

To address Homeland Security needs for decontamination, the U.S. Environmental Protection Agency (EPA) established an Interagency Agreement with the U.S. Army Edgewood Chemical Biological Center (ECBC) to take advantage of ECBC's extensive expertise and specialized research facilities for the decontamination of surfaces contaminated with chemical and biological (CB) warfare agents. The National Homeland Security Research Center (NHSRC) formed a collaboration with ECBC in a mutual leveraging of resources, expanding upon ECBC's on-going programs in CB decontamination to more completely address the parameters of particular concern for decontamination of indoor surfaces in buildings following a terrorist attack using CB agents, toxic industrial chemicals or materials. In the context of decontamination, the contaminants of interest are those that can persist on indoor surfaces, leading to continuing chance of exposure long after the contamination occurs. TheVHP and ClO_2 are decontamination technologies that have been used to decontaminate indoor surfaces contaminated with anthax spores and show potential for use in decontaminating indoor surfaces contaminated by some chemical agents. The systematic decontamination was specifically focused on decontamination of the building environment for purposes of restoring a public building to a usable state after a terrorist contamination episode. Systematic testing of decontamination technologies generated objective performance data so building and facility managers, first responders, groups responsible for building decontamination, and other technology buyers and users can make informed purchase and application decisions.

Because building interiors may contain large surfaces composed of complex materials, the Material Compatibility effort was designed to determine how the decontaminant vapors impacted building materials within an enclosed building interior space. The objective of this study was to establish and conduct laboratory test procedures to determine to what degree building materials were affected after decontamination using VHP and ClO_2. The building interior materials used for testing were a subset of the variety of structural, decorative, and functional materials common to commercial office buildings, regardless of architectural style and age. The building materials encompassed a variety of material compositions and porosities; the materials studied included unpainted concrete cinder block, standard stud lumber (wood 2 x 4 in., fir, type-II), latex-painted ½ in. gypsum wallboard, acoustical ceiling suspension tile, primer-painted structural steel, and carpet. The Material Compatibility studies also investigated material(s) related to electrical breaker connections. The physical appearance was documented by visual inspection of the test material. The physical properties of the building materials were measured using standardized ASTM and UL test methods. Specialized chemical testing was conducted to determine if chemical changes occurred in select building materials.

The process for exposing the building material samples to ClO_2 and results for the material demand study are documented in a separate report titled "Material Demand Studies: Materials Sorption of Chlorine Dioxide," by Philip W. Bartram, et al.

4. EXPERIMENTAL PROCEDURES

The Material Compatibility testing was conducted in compliance with the Quality Assurance Project and Work Plan[1] developed under the Quality Management Plans[2,3] and EPA E4 quality system requirements.[4–7]

4.1 Coupon Preparation

Test coupons were prepared in accordance with the ASTM testing requirements for the Material Compatibility testing and were assigned a unique identifier as shown in Appendix A. The coupons were cut from stock material in accordance with the procedure in Appendix B of the Quality Assurance Project Plan (QAPP)[8], which has been reproduced as Appendix B of this report. Coupons were prepared by obtaining a large enough quantity of material that multiple test samples could be obtained with uniform characteristics (e.g., test coupons were all cut from the interior rather than the edge of a large piece of material). The building materials that were studied, including supplier and coupon dimensions, are provided in Table 1 and Figure 1.

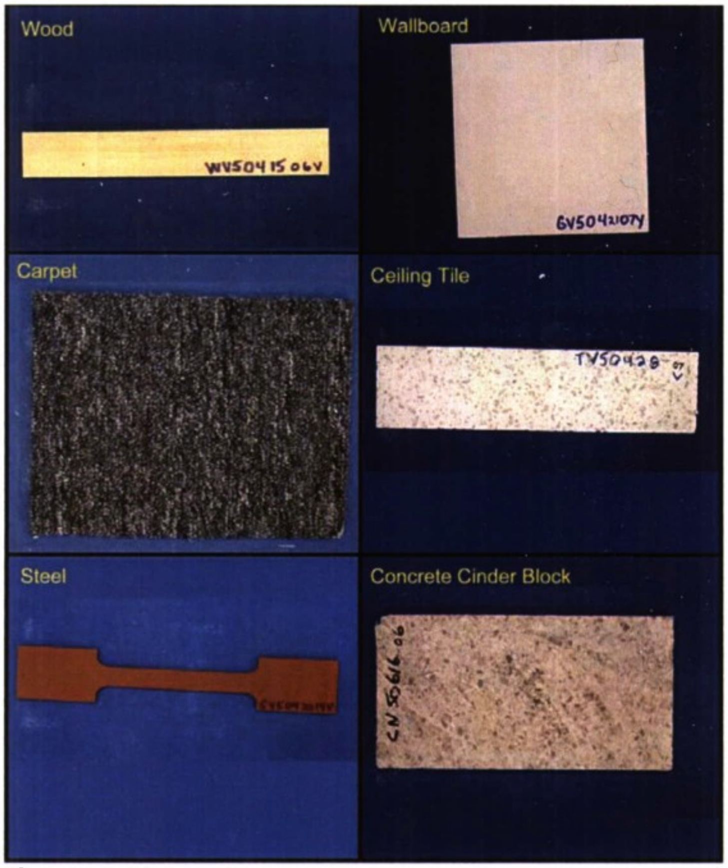
Table 1. Representative building interior material list.

Material	Code	Supplier	Length	Width	Thickness
Structural Wood, Fir, Type-II	W	Home Depot	10 ± 0.062 in.	1.5 ± 0.062 in.	0.5 ± 0.03125 in.
Latex-Painted Gypsum Wallboard	G	Home Depot	6 ± 0.062 in.	6 ± 0.062 in.	0.5 ± 0.062 in.
Concrete	C	York Supply	4 ± 0.5 in.	8 ± 0.5 in.	1.5 ± 0.1875 in.
Carpet	R	Home Depot	6 ± 0.5 in.	8 ± 0.125 in.	N/A
Painted Structural Steel	S	Specialized Metals	12 ± 0.062 in.	2 ± 0.0625 in. 0.75 ± 0.062 in.	0.25 ± 0.00781 in.
Ceiling Suspension Tile, Acoustical	T	Home Depot	12. ± 0.125 in.	3 ± 0.062 in.	0.56 ± 0.062 in.
Circuit Breakers	B	Home Depot	N/A	N/A	N/A

Chain-of-custody (COC) cards were used to ensure that the test coupons were traceable thoughout all phases of testing. The test coupons were measured and visually inspected prior to testing.

Coupons were measured to ensure that the test coupon was within the acceptable tolerances (Appendix B). Coupons were visually inspected for defects and/or damage. Coupon measurements and visual inspection were recorded on the COC card. Coupons that were not within the allowable size tolerances and/or damaged were discarded. Each coupon was assigned a unique identifier code that matches the coupon with the sample, test parameters, and sampling scheme (Appendix A). The unique identifier code was recorded on the COC form. The COC cards followed each sample from exposure testing though Material Compatibility testing to disposal.

The Material Compatibility studies also investigated materials related to electrical breaker connections, such as intact circuit breakers and component metals aluminum, copper, and steel. The circuit breakers were one-pole circuit breakers (HOM120, 2400 W, 120/240 V, 20 A).



*Coupons are not shown to scale

Figure 1. Representative photograph of the test coupons.

4.2 Coupon Exposure: Wood, Wallboard, Ceiling Tile, Steel, Carpet, and Concrete Cinder Block

The process for exposing the building material samples to ClO_2 and results for the material demand study are documented in a separate report titled "Material Demand Studies: Materials Sorption of Chlorine Dioxide," by Phil Bartram, et al. This testing followed the operating procedures discussed with the sponsor and is not specific to any particular vendor. A brief overview of the exposure process is provided in this section. The Material Demand report contains the detailed test information and results.

The coupons were placed in the exposure chamber. The chamber was conditioned to achieve the target relative humidity (RH) or 75% and target temperature of 75 °F. The vapor generator was operated to maintain the chamber concentration within specified ranges. The full-target concentration was 2000 ppm ClO_2 for 6 h for a total concentration time (CT) value of 12,000 ppm-hs. The half-target concentration was 1000 ppm ClO_2 for 12 h for a total CT value of 12,000 ppm-hs. The ClO_2 tests were conducted with a turnover rate of approximately one air exchange per h due to the relative stability of ClO_2 to mimic actual treatment conditions. Aeration of the chamber was conducted following the decontamination phase (exposure period). Aeration of chamber continued until the vapor concentration fell to/below the levels required by the Risk Reduction Office to ensure safe operation for personnel. The coupons remained in the chamber until aeration was complete. Control samples were prepared using the same procedure as the test runs except with only air (no-fumigant) though the chamber. Three replicate runs were performed for each sample at each condition. The samples were removed from the chamber and marked with unique sample identifier codes and visually examined.

4.3 Coupon Exposure: Circuit Breakers

The circuit breakers (HOM220, Home Depot) were placed in the exposure chamber and exposed to fumigant as the other building materials discussed in Section 4.2. After exposure to the decontaminant, the circuit breakers were stored in a fume hood for 2 days, and then placed in storage under load for 3 months. Each set of circuit breakers was inserted into an electrical box (8 spaces, 16 circuits, 100 amp max from square D, Home Depot No. 577-340). The circuit breaker box was wired with 12 gauge, 20 amp wire into the 120 V outlet. Each circuit breaker was wired in series with an electrical lamp (s513e) with an outlet box (s110e) manufactured by Thomas & Bretts (Home Depot No. c214477 and b214426, respectively). The load in each lamp was a Phillips 40 watt light bulb (Philips and Sylvania, Home Depot). Current was applied to the circuits and monitored. At the end of 90 days, the circuit breakers were tested to determine the effect of ClO_2.

4.4 Visual Inspection

The coupons were visually inspected and digitally photographed upon removal from the chamber. Visual inspection of the coupon surfaces was conducted though side-by-side comparison of the decontaminated test surface and fresh coupons of the same test material. The testing staff looked for changes such as discoloration, blistering, warping, and peeling on the test coupon compared to the fresh coupon. After the visual inspection was completed, the coupon custody was transferred to the Material Compatibility Technical Leader for the 3 month aging period and Material Compatibility testing. The coupons were examined again at the time of the material testing and the visual appearance recorded on the data test forms. If the coupon had dramatic changes compared to a fresh coupon, then the coupon was photographed. Representative photographs of each material type are provided in the appendices to this report.

4.5 Coupon Aging

The Material Compatibility studies were conducted using the coupons from the Material Demand study. The coupons were aged for a minimum of 90 days following exposure to decontaminant and prior to Material Compatibility testing. The coupons were placed in open containers and stored under ambient conditions. The open container arrangement allowed aging of the coupons in conditions mimicking real world aging.

4.6 Data Review and Technical Systems Audits

The approved Material Compatibility QAPP specified procedures for the review of data and independent technical system audits. All data were peer reviewed within 2 weeks of collection. The project quality manager (or designee) was required to audit at least 10% of the data collected. The project quality manager (or designee) performed four technical system audits over the course of testing. A technical system audit is a thorough, systematic, on-site qualitative audit of the facilities, equipment, personnel, training, procedures, recordkeeping, data validation, data management and reporting aspects of the system.

4.7 Physical Testing

An Instron model 5582 was used for the physical property testing. The Instron is a universal testing machine capable of performing tensile, compression, shear, peel, and flexural tests on most materials and components. Each material subsection contains a photograph of the coupon loaded into the test apparatus. The Instron model 5582 specifications are listed in Table 2.

Table 2. Instron model 5582 specifications.

Feature	Units	Value
Load Capacity	kN	100
	kgf	10000
Maximum Speed	mm/min	500
Minimum Speed	mm/min	0.001
Maximum Force at Full Speed	kN	75
Maximum Speed at Full Load	mm/min	250
Return Speed	mm/min	600
Position Control Resolution	mm	0.06
Total Crosshead Travel	mm	1235
Total Vertical Test Space	mm	1309
Height	mm	2092
Width	mm	1300
Depth	mm	756
Weight	kg	862

4.8 Statistical Analyses

The data from the Material Compatibility testing phase of the Systematic Decontamination program was subjected to a statistical analysis to determine if the differences observed among the various test sets were merely the result of random variations in test data, or represented actual differences in the performance of the materials as a result of exposure to fumigation chemicals.

Methods were used from both the statistical analysis functions embedded within the Microsoft Excel software, and Practical Statistics for Analytical Chemists, Robert L. Anderson, © 1987 by Van Nostrand Reinhold Company.

First, the individual coupon sets were tested to see if there were statistical outliers that could be eliminated from the data. The Q-Test for outliers was first used to identify potential outliers within a test set. Then the test group of coupons that had undergone similar treatment (controls, half-target, or full-target exposures) was tested. If an outlier identified in the individual coupons was also picked out in the test group analysis, the outlier was eliminated and the statistics (averages and standard deviations) recalculated. However, if the specific data point was not identified as an outlier by both tests, it was retained in the study. Once statistical outliers had been eliminated, the test groups were analyzed to determine if they were significantly different statistically—that is, to determine if the treatment with the chosen fumigant had a detectable effect on the sample.

The primary test used was the Welch's *t* test; the two-tailed, heteroscedastic test was used for the analysis. Welch's *t* test values were calculated to compare the test groups and results are reported for 95% level of confidence. The reported percent level of confidence indicated the confidence of the two sample groups being compared were, in fact, different, and represented truly different samples. A 95% level of confidence indicated that there was a 5% chance (one chance in 20) that the two samples were, in fact, sub-parts of the same population. If a comparison determined that a sample was significantly different at the X% level of confidence, it was also significantly different at any lower level of confidence.

Determination that a control and exposed sample were statistically different implied that the treatment had some detectable effect on the material. Statistically different results did not imply that the material failed as a result of treatment, unless the material no longer met specifications. In some cases, measured values varied by several percent; however, there was no statistically detectible difference. It cannot be assumed that this difference was real unless the difference was statistically detected (e.g., by a Welch's *t* test).

4.9 Chemical Testing by Ion Chomatography

The Systematic Decontamination program also included additional ClO_2 tests of metals commonly used in electrical applications. Metal samples were exposed to ClO_2 and analyzed for the direct analysis of chloride, chlorite, chlorate, and perchlorate anions in aqueous ClO_2 decontamination matrices using an ion chomatograph (IC) with conductivity detection. The results of this study are discussed in Section 12 of this report.

4.10 Post Fumigation Inspection

The coupons were visually inspected prior to fumigation, immediately after fumigation and after storage at time of material testing. Carpet coupons were inspected for any frayed tufts, pulled loops, and other noticeable defects. Concrete coupons were inspected for cracks, chips—particularly at the corners, any raised ridge sections, and other noticeable defects. Steel coupons were inspected for any ridged sections on the small I-beam cross section, rust, peeling paint, and any other noticeable defects. Tile coupons were inspected for crushed corners and edges, and any other noticeable defect. Wallboard coupons were inspected for any damage to the paper section, as well as any other noticeable defects. Wood coupons were inspected for any knots, missing knots, splitting, and other noticeable defects. The inspections were compared to the initial inspections. No differences were observed for any of the coupons after ClO_2 exposure and aging, compared to before ClO_2 exposure.

5. EVALUATION OF STRUCTURAL STEEL

5.1 Introduction

The effects of ClO_2 on the physical integrity of steel were investigated using the tension test as described in ASTM test method A370-03a "Standard Test Methods and Definitions for Mechanical Testing of Steel Products," Sections 5–13. The tension test was used to determine the integrity of steel coupons exposed to vaporous decontaminant compared to unexposed (control) steel coupons.

5.2 Sample Preparation and Testing

The steel samples were removed from storage, visually inspected, and measured to confirm samples were within coupon specifications listed in Appendix A. The coupons from chamber positions 1, 4, 7, 10, and 16 were selected for testing; the coupons were selected to obtain representation thoughout the test chamber. The samples were used "as is" without any additional preparation. The testing was conducted in accordance with the ASTM test method A370-03a. The Instron fixture for the steel test was installed prior to testing. The Instron universal testing machine operation and calibration verification was conducted by suspending a certified weight from the fixture and recording the weight. Three sets of five coupons were tested for each concentration (target and half-target) and four sets were tested for the controls (0 ppm). The load required to rupture the steel coupons was measured in Newtons (N). The tensile strength is the maximum tensile stress that a material is capable of sustaining and is calculated by dividing amount of force required to rupture a specimen by the specimen cross-sectional area. No precision or bias requirements were established for this test method. The results of control coupons were compared against decontaminant exposed samples. A statistical analysis of the data was conducted to determine if the decontaminant exposed steel coupon results were statistically different compared to the control steel coupons.

5.3 Results

The coupons were stored for at least 90 days after fumigation. The actual number of storage days was based on the arrival of the Instron fixtures for testing. The coupons for a particular fumigation trial were studied after a similar number of days in storage. A photograph of a representative steel sample before and after testing is provided in Figure 2. The load required to rupture the steel coupons, the tensile strength results, and number of days in storage before testing values are provided in Table 3.

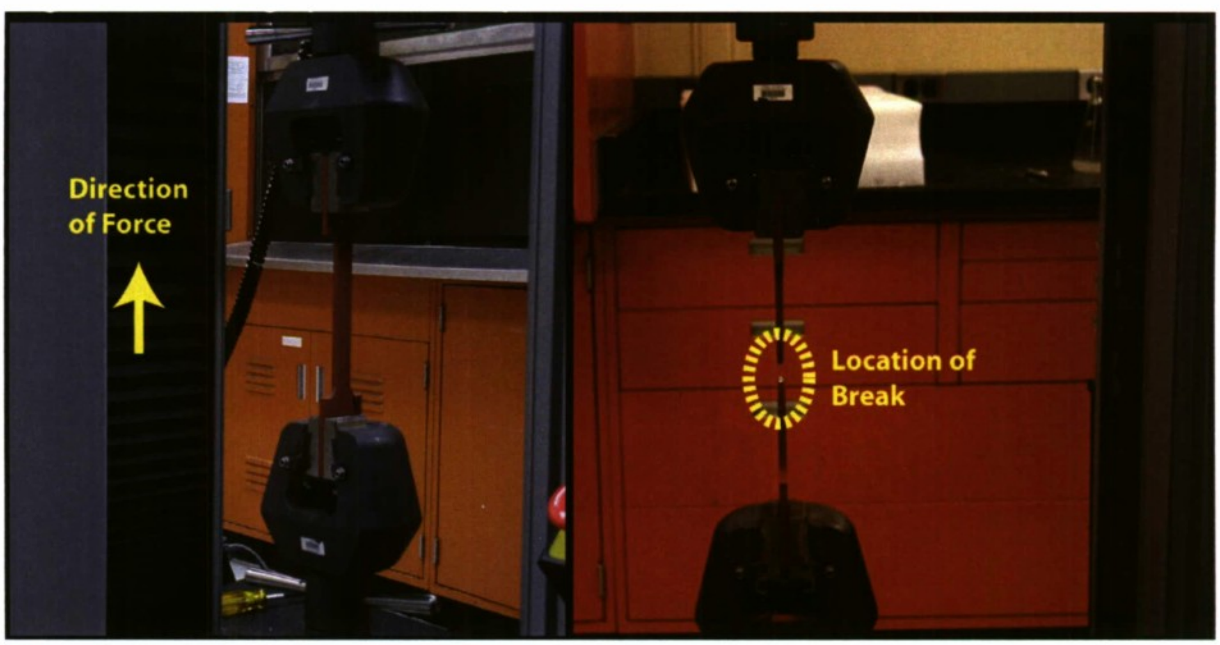


Figure 2. Steel coupon test representative photograph.

Table 3. ClO_2 steel coupon test results.

Maximum Load	0 ppm Control Samples Tension Test Results (N)				1000 – 1250 ppm Half-Target Concentration Results (N)			2000 – 2500 ppm Full-Target Concentration Results (N)		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon Set ID	SN5 0302	SSN5 1018	SSN5 0525	SN5 0228	SSD5 0928	SSD5 1004	SSD5 1005	SSD5 0919	SSD5 0921	SSD5 0927
Coupon 1	60616	60025	61501	61175	62076	63593	62285	60049	63305	64290
Coupon 2	60916	58303	61041	61559	63920	63181	64543	64989	62608	63527
Coupon 3	61191	63318	62511	60806	61906	58890	63163	60248	62549	59927
Coupon 4	60890	59384	60302	60731	61904	62222	59911	62529	63729	63872
Coupon 5	61049	64803	61698	60900	64228	61673	61122	61831	63135	62932
Test Avg	60932	61167	61410	61034	62807	61912	62205	61929	63078	62909
Std Dev	214	2763	817	338	1164	1852	1791	2006	516	1740
Test Set Avg ± Std Dev	61136 ± 1347				62308 ± 1560			62639 ± 1538		
Tensile Strength	**Control Samples Tensile Strength Results (N/mm²)**				**Half-Target Concentration Results (N/mm²)**			**Full-Target Concentration Results (N/mm²)**		
Coupon Set ID	SN5 0302	SSN5 1018	SSN5 0525	SN5 0228	SSD5 0928	SSD5 1004	SSD5 1005	SSD5 0919	SSD5 0921	SSD5 0927
Coupon 1	561	527	569	537	575	558	546	556	586	536
Coupon 2	564	540	535	570	561	554	538	570	522	529
Coupon 3	567	555	548	563	573	545	538	570	522	529
Coupon 4	564	550	558	562	543	546	526	579	560	560
Coupon 5	536	540	571	534	563	541	536	542	554	552
Test Avg	558	542	557	553	563	549	524	561	560	541
Std Dev	13	11	15	17	13	7	29	14	25	15
Test Set Avg ± Std Dev	553 ± 14				545 ± 24			554 ± 20		
Number of Days in Storage	**Control Samples (Days)**				**1000 – 1250 ppm Half-Target Concentration (Days)**			**Full-Target Concentration (Days)**		
Coupon Set ID	SN5 0302	SSN5 1018	SSN5 0525	SN5 0228	SSD5 0928	SSD5 1004	SSD5 1005	SSD5 0919	SSD5 0921	SSD5 0927
Days	103	159	96	98	173	168	167	220	218	181
Test Set Avg ± Std Dev	114 ± 27				169 ± 3			206 ± 19		

Note: The cell highlighted in orange indicates that the data point was statistically identified as an outlier within its test set, but not within the test group (four control groups); therefore, the value was retained.

5.4 Discussion

The steel studied was an A572 Grade 50 high strength, structural steel. The minimum tensile strength requirement was 450 N/mm^2. The control coupons and ClO_2-exposed coupons met this minimum specification.

Of the 50 coupons tested in this portion of the program, only one generated a result that could be rejected as a statistical outlier from within its individual test set at the Q=0.99 level of confidence—Coupon 5 from test set SN50302, which had a tensile strength value significantly below the others in its test set. Within test groups (control samples, half-target concentration samples, and full-target concentration samples), similar statistical analysis showed that none of the coupon sets could be eliminated as statistical outliers. Therefore, coupon 5 was retained for this analysis.

The values for the maximum load for the steel coupons were determined to be 61136 ± 1347 N for the control samples, 62308 ± 1560 N for the half-target samples, and 62639 ± 1538 N for the full-target coupons. The value for the tensile strength of the steel coupon is the maximum load (Newton) divided by the cross-sectional area (mm^2) of the coupon at the break point. The values for the tensile strength of the steel coupons were calculated to be 553 ± 14 N/mm^2 for the control coupons, 545 ± 24 N/mm^2 for the half-target coupons, and 554 ± 20 N/mm^2 for the full-target coupons.

When considering the data from the test groups of coupons, the average maximum load values for the ClO_2 exposed coupons differ by less than 3% from the control samples, and the tensile strengths vary by about 1%. The Welch's *t* test was used to determine if any of the groups of samples were statistically different from the others. The tensile strength results showed that there was no statistical difference between the controls, half-target, or full-target concentration samples at the 95% confidence level. The average maximum load values for the half-target or full target coupon samples, however, were statistically different from the control samples at the 95% confidence level. This was a result of slight difference in the cross-sectional areas between the groups that were still within the target tolerance values for quality control and not due to an affect of the fumigation process.

6. EVALUATION OF GYPSUM WALLBOARD

6.1 Introduction

The effects of ClO_2 on the physical integrity of gypsum wallboard were investigated using the nail pull resistance test method B as described in ASTM Test C473-03 "Standard Test Methods for Physical Testing of Gypsum Panel Products," Section 13. The test measures the ability of the wallboard to resist nail pull-though by determining the load required to push a standard nail though the wallboard. The ASTM test was used to determine the integrity of the gypsum wallboard coupons exposed to vaporous decontaminant compared to unexposed (control) gypsum wallboard coupons.

6.2 Sample Preparation and Testing

The gypsum wallboard samples were removed from storage, visually inspected, and measured. The coupons from chamber positions 1, 2, 4, 5, and 7 were selected for testing; the coupons were selected to obtain representation thoughout the test chamber. The samples were brought to moisture equilibrium such that the weight of the sample did not change by more than 0.2% on successive weighings at a minimum interval of 2 h. The sample preparation was conducted within a temperature range of 15–25 °C and an RH of 48–75%. The testing was conducted in accordance with ASTM test method C473-03. The Instron fixture for the gypsum wallboard test was installed prior to testing. The Instron universal testing machine operation was verified by suspending a certified weight from the fixture and recording the weight. Three sets of five coupons were tested for each concentration (full-target and half-target) and four sets were tested for the controls (0 ppm). The force required to drive a nail shank though the wallboard coupons was measured in N. The ASTM method indicates that any coupon measurement in the series that varies 15% more than the average needs to be discarded. If 15% of the coupons deviate from the average, the method states that the test will be repeated. No additional precision or bias requirements were determined for this test in accordance with the ASTM method. The results of control coupons were compared against decontaminant-exposed samples. A statistical analysis of the data was conducted to determine if the decontaminant-exposed coupon results were statistically different compared to the control coupons.

6.3 Results

The coupons were stored for at least 90 days. The actual number of storage days was based on the arrival of the Instron fixture for testing. The coupons for a particular fumigant were studied at the same number of days. A photograph of a gypsum wallboard sample before and after testing (i.e., holes) is provided in Figure 3. Table 4 provides the load required to push the nail though the wallboard coupons and number of days in storage before testing values.

6.4 Discussion

The wallboard tension test results were analyzed for potential statistical outliers using the Q-Test and for differences between the control and exposed samples using Welch's *t* test. Although there was a great deal of scatter in the data (the data ranged from 37.0–73.5 N and the standard deviations of the results were between 9–21% of the mean value within the various test groups); therefore, none of the individual coupons were determined to be outliers at the Q=0.99 confidence level.

The average tension test results are 50.2 ± 9.7 N for the control group, 59.4 ± 12.1 N for the half-target group, and 56.7 ± 5.4 N for the full-target group. The differences were not determined to be statistically significant at the 95% confidence interval as determined by the Welch's *t* Test. Therefore, exposure to the ClO_2 fumigation process appeared to have no impact on the wallboard with respect to the ASTM tension test used for analysis.

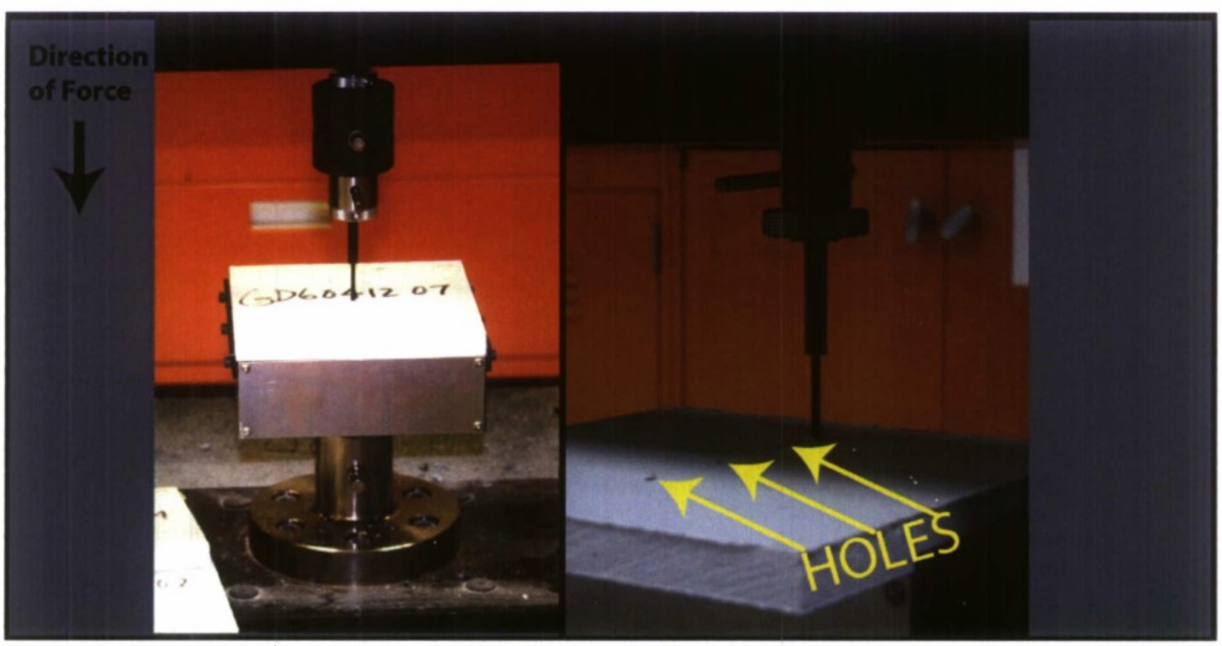


Figure 3. Representative photograph of the gypsum wallboard coupon test.

Table 4. Gypsum wallboard coupon test results for maximum load.

Force	0 ppm Control Samples Tension Test Results (N)				1000 – 1250 ppm Half-Target Concentration Results (N)			2000 – 2500 ppm Full-Target Concentration Results (N)		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon Set ID	GN5 0316	GN5 0301	GN5 0518	GN5 0512	GD6 0418	GD6 0420	GD6 0424	GD5 0417	GD6 0412	GD6 0413
Hole 1	64.2	55.1	43.4	45.3	61.29	56.33	47.96	57.1	40.2	67.88
Hole 2	62.3	44.1	37.3	4106	52.19	47.72	50.49	69.4	49.6	65.32
Hole 3	60.6	48.8	37.0	47.2	65.02	62.27	63.78	72.1	43.9	70.30
Hole 4	71.0	54.3	38.3	52.0	55.19	55.18	57.92	56.4	47.8	69.5
Hole 5	61.0	45.8	43.8	51.2	58.36	57.50	59.45	67.4	41.0	73.48
Test Avg	63.8	49.6	39.9	47.5	58.4	55.8	55.92	64.5	44.5	69.3
Std Dev	4.2	5.0	3.4	4.3	5.0	5.3	6.5	7.3	4.1	3.0
Test Set Avg ± Std Dev	50.2 ± 9.7				56.7 ± 5.4			59.4 ± 12.1		
Days in Storage	175	190	349	355	157	155	151	157	161	163

7. EVALUATION OF ACOUSTICAL CEILING TILE

7.1 Introduction

The effects of ClO_2 on the physical integrity of ceiling tile were investigated using the transverse strength test as described in ASTM Test C367-99 "Standard Test Methods for Strength Properties of Prefabricated Architectural Acoustical Tile or Lay-In Ceiling Panels," Sections 1, 3–5 and 21–29. The test measures the force required to cause the tile to break. The ASTM test was used to determine the integrity of the ceiling tile coupons exposed to vaporous decontaminant compared to unexposed (control) ceiling tile coupons.

7.2 Sample Preparation and Testing

The acoustical ceiling tile samples were removed from storage, visually inspected, and measured. The samples were brought to moisture equilibrium such that the weight of the sample did not change by more than 1% on successive weighings at a minimum interval of 2 h. The sample preparation was conducted within a temperature range of 18–24 °C and a relative humidity of 48–75%. The testing was conducted in accordance with the ASTM test method C367-99. The Instron fixture for the ceiling tile test was installed prior to testing. The Instron universal testing machine operation was verified by suspending a certified weight from the fixture and recording the weight. For each test, the coupons from chamber positions 1 though 8 were selected for testing; this selection consisted of all coupons placed in the chamber during a single fumigation trial. Three sets of four machine direction coupons and four cross-machine direction coupons were tested for each concentration (full-target and half-target) and four sets were tested for the controls (0 ppm) for each direction. The load required to break the ceiling tile coupons was measured in N. Figure 4 shows a photograph of a coupon loaded into the Instron for the machine and cross-machine direction tests. No precision or bias requirements were established for this test method. The results of control coupons were compared to decontaminant-exposed tiles. A statistical analysis of the data was conducted to determine if the decontaminant-exposed coupon results were statistically different compared to the control coupons.

The Modulus of Rupture (MOR) was calculated according to the test method using Equation 1. P is the maximum load, N (lbf). L is the length of span, mm (in.). Variables b and d are the specimen width and thickness, respectively in millimeters.

Equation 1

$$\text{MOR units N/mm}^2\ (\text{lb/in}^2) = \frac{3 \times P \times L}{2 \times b \times d^2}$$

Figure 4. Representative photograph of the acoustical ceiling tile coupon test.

7.3 Results

The coupons were stored for at least 90 days. The actual number of storage days was based on the arrival of the Instron fixture for testing. The coupons for a particular fumigation trial were studied at the same number of days. Figure 5 provided a photograph of a representative ceiling tile sample before and after testing. Table 5 provides the load required to break the ceiling tile coupons, the ceiling tile coupon MOR results, and number of days in storage.

7.4 Discussion

Ceiling tile coupons were tested in two directions—with the mandrel parallel to the axis of the test machine (hereafter referred to as machine direction), and with the mandrel perpendicular to the axis (cross-machine). Coupons were tested for maximum load, which was then used to calculate the MOR for each coupon.

For the machine direction tests, the maximum load values were 37.17 ± 5.72 N for the control samples, 39.90 ± 3.89 N for the half-target coupons, and 36.03 ± 3.56 N for the full-target coupons. The MOR values for the machine direction tests were 0.86 ± 0.13 N/mm² for the control samples, 0.92 ± 0.09 N/mm² for the half-target coupons, and 0.83 ± 0.08 N/mm² for the full-target coupons.

For the cross-machine tests, the maximum load values were 29.95 ± 5.68 N for the control samples, 32.52 ± 3.68 N for the half-target coupons, and 26.82 ± 2.22 N for the full-target coupons. The MOR values for the cross-machine direction tests were 0.69 ± 0.13 N/mm² for the control samples, 0.76 ± 0.09 N/mm² for the half-target coupons, and 0.62 ± 0.05 N/mm² for the full-target coupons.

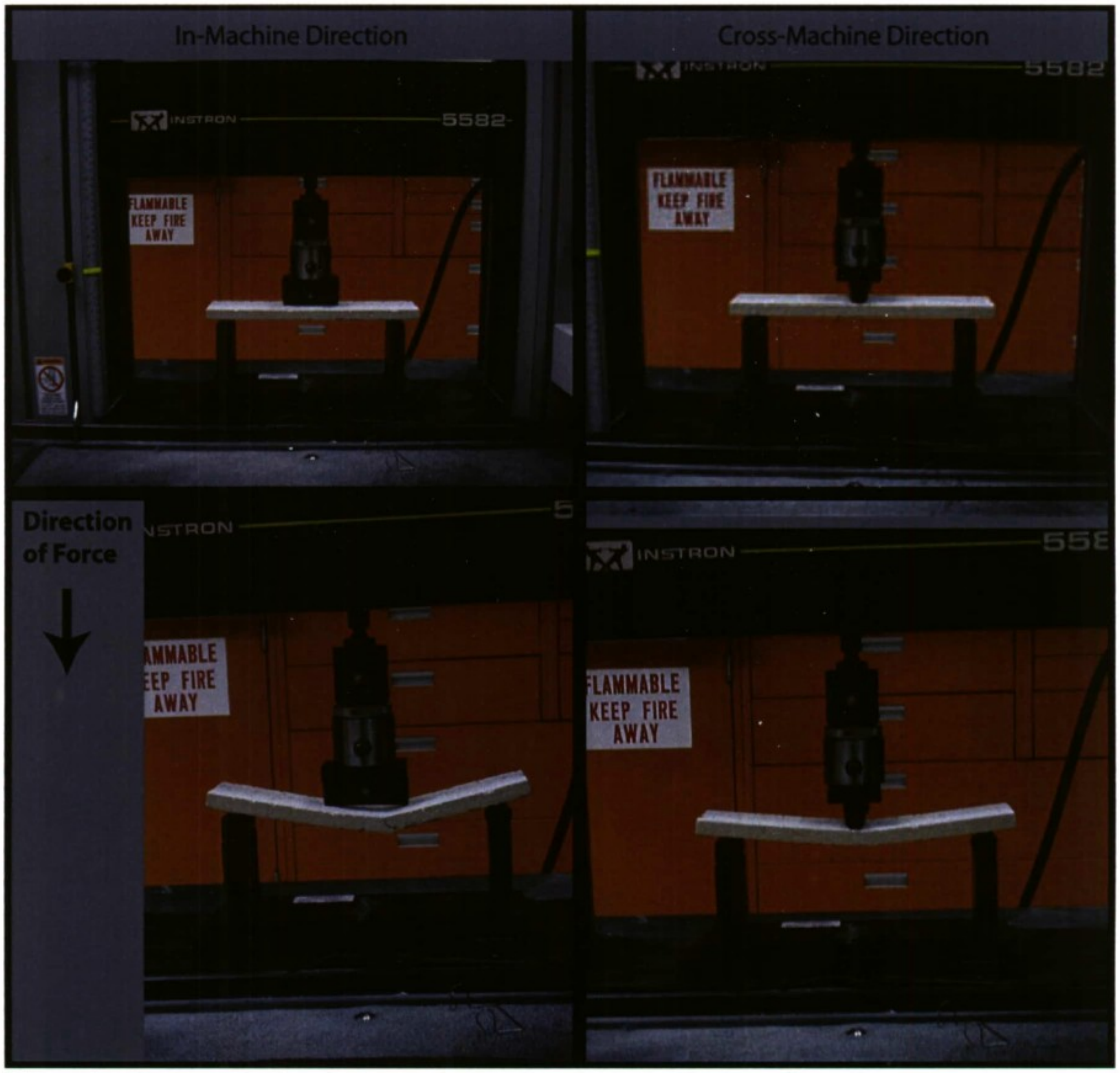


Figure 5. Photograph showing acoustical ceiling tile end of test configuration.

A statistical analysis of the individual test results was conducted to detect potential statistical outliers (Q-Test) and determine if there are any differences between the control and exposed samples (Welch's *t* Test).

Only one of the individual coupons was determined to be an outlier at the Q=0.99 confidence level (Coupon 3 of test set TD60328, half-target concentration) within its own test set. Because the machine direction maximum load of this coupon was significantly different statistically from others in the test set, the value for the MOR for that coupon was also a statistical outlier. However, when compared to all the other tests and coupons in the test group (half-target in-line), the coupon was not significantly different, and therefore, could not be rejected.

The half-target exposure coupons produced slightly higher maximum load values and higher modulus of rupture values (on average) than either the control or full-exposure coupons for the machine direction and the cross-machine tests. However, at the 95% confidence level there were no statistical differences between the control samples and either the half-target or full-target concentration samples.

Table 5. ClO_2 coupon test results for tile.

Maximum Load – Machine Direction	**0 ppm Control Samples Tension Test Results (N)**				**1000 – 1250 ppm Half-Target Concentration Results (N)**			**2000 – 2500 ppm Full-Target Concentration Results (N)**		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon Set ID	TN5 1024	TN5 0223	TN5 0517	TN5 0519	TD6 0301	TD6 0329	TD6 0328	TD6 0228	TD6 0223	TD6 0227
Coupon 1	37.10	31.27	51.49	38.82	44.48	42.80	41.17	34.31	40.08	32.58
Coupon 2	39.63	29.80	32.19	43.15	41.09	42.80	41.13	41.68	35.96	32.87
Coupon 3	33.44	30.82	36.85	41.70	34.79	36.71	37.14	36.69	36.75	33.19
Coupon 4	36.73	34.54	40.48	42.76	31.88	43.76	41.01	38.21	40.03	29.99
Coupon 5		31.19								
Test Avg	36.73	31.52	40.25	41.61	38.06	41.52	40.11	37.72	38.21	32.16
Std Dev	2.54	1.78	8.22	1.96	5.75	3.24	1.98	3.09	2.16	1.47
Test Set Avg ± SD	37.17 ± 5.72				39.90 ± 3.89			36.03 ± 3.56		

Modulus Rupture – Machine Direction	**Control Samples Tensile Strength Results (N/mm²)**				**Half-Target Concentration Results (N/mm²)**			**Full-Target Concentration Results (N/mm²)**		
Coupon Set ID	TN510 24	TN502 23	TN505 17	TN505 19	TD603 01	TD603 29	TD603 28	TD602 28	TD602 23	TD602 27
Coupon 1	0.86	0.72	1.19	0.91	1.03	0.98	0.95	0.79	0.93	0.75
Coupon 2	0.92	0.69	0.75	1.00	0.95	0.99	0.95	0.97	0.83	0.76
Coupon 3	0.77	0.71	0.85	0.95	0.76	0.85	0.86	0.85	0.85	0.77
Coupon 4	0.85	0.80	0.94	0.99	0.75	1.01	0.95	0.88	0.93	0.69
Coupon 5		0.72								
Test Avg	0.85	0.73	0.93	0.96	0.87	0.96	0.93	0.87	0.88	0.74
Std Dev	0.06	0.04	0.19	0.04	0.14	0.07	0.05	0.07	0.05	0.03
Test Set Avg ± SD	0.86 ± 0.13				0.92 ± 0.09			0.83 ± 0.08		

Table 5. ClO_2 coupon test results for tile (continued).

Maximum Load – Cross Machine	**Control Samples Tension Test Results (N)**				**Half-Target Concentration Results (N)**			**Full-Target Concentration Results (N)**		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon Set ID	TN510 24	TN502 23	TN505 17	TN505 19	TD603 01	TD603 29	TD603 28	TD602 28	TD602 23	TD602 27
Coupon 1	33.47	22.21	32.84	37.52	24.33	33.34	33.62	32.06	26.95	25.00
Coupon 2	21.19	19.57	33.67	36.24	37.33	35.45	32.80	25.40	27.75	25.35
Coupon 3	26.77	26.49	34.32	32.36	31.66	30.79	33.84	25.74	25.37	30.18
Coupon 4	26.80		32.11	33.72	37.16	31.99	27.96	26.88	24.97	26.23
Test Avg	27.06	22.76	33.24	34.96	32.62	32.89	32.06	27.52	26.26	26.69
Std Dev	5.02	3.49	0.96	2.34	6.12	2.00	2.77	3.09	1.31	2.38
Test Set Avg ± SD	29.95 ± 5.68				32.52 ± 3.68			26.82 ± 2.22		

Modulus of Rupture – Cross Machine	**Control Samples Tensile Strength Results (N/mm²)**				**Half-Target Concentration Results (N/mm²)**			**Full-Target Concentration Results (N/mm²)**		
Coupon Set ID	TN5 1024	TN5 0223	TN5 0517	TN5 0519	TD6 0301	TD6 0329	TD6 0328	TD6 0228	TD6 0223	TD6 0227
Coupon 1	0.78	0.51	0.76	0.87	0.56	0.77	0.78	0.74	0.62	0.58
Coupon 2	0.49	0.45	0.78	0.84	0.85	0.82	0.76	0.59	0.64	0.59
Coupon 3	0.62	0.61	0.80	0.75	0.73	0.71	0.78	0.60	0.59	0.70
Coupon 4	0.62		0.74	0.78	0.86	0.86	0.65	0.62	0.58	0.61
Test Avg	0.63	0.52	0.77	0.81	0.75	0.79	0.74	0.64	0.61	0.62
Std Dev	0.12	0.08	0.02	0.05	0.14	0.06	0.06	0.07	0.03	0.06
Test Set Avg ± SD	0.69 ± 0.13				0.76 ± 0.09			0.62 ± 0.05		

Number of Days in Storage	**Control Samples Days**				**Half-Target Concentration Days**			**Full-Target Concentration Days**		
Coupon Set ID	TN5 1024	TN5 0223	TN5 0517	TN5 0519	TD6 0301	TD6 0329	TD6 0328	TD6 0228	TD6 0223	TD6 0227
Days in Storage	200	203	307	305	204	176	177	205	210	206
Test Set Avg ± SD	254 ± 60				186 ± 16			207 ± 3		

Note: Orange highlighted cells indicate that the data point was statistically determined to be an outlier within its test set, but not within the test group (four control groups); therefore, the value was retained.

8. EVALUATION OF CARPET

8.1 Introduction

The effects of ClO_2 on the physical integrity of loop pile carpet fibers were investigated using ASTM Test C1335-03 "Standard Test Method for Tuft Bind of Pile Yarn Floor Coverings." The method determines the force required to pull out a tuft of a pile yarn from a floor-covering sample. The ASTM test was used to determine the integrity of the loop pile carpet fibers exposed to vaporous decontaminant compared to unexposed (control) loop pile carpet fibers.

8.2 Sample Preparation

The carpet samples were removed from storage, visually inspected, and measured. The coupons from chamber positions 1, 3, 4, 5, and 7 were selected for testing; the coupons were selected to obtain representation throughout the test chamber. The samples were brought to moisture equilibrium such that the weight of the sample did not change by more than 0.2% on successive weighings at a minimum interval of 2 h. The sample preparation was conducted within a temperature range of 15–24 °C and a relative humidity of 48–75%. The testing was conducted in accordance with the ASTM test method D1335-03. The Instron fixture for the carpet test was installed prior to testing. The Instron universal testing machine operation and calibration verification was conducted by suspending a certified weight from the fixture and recording the weight. Three sets of five coupons were tested for each concentration (target and half-target); the coupons were selected to obtain representation throughout the test chamber (Figure 6). The load required to pull a carpet loop from the binding was measured in N. No bias requirements were established for this test method. The results of control coupons were compared to decontaminant exposed samples. A statistical analysis of the data was conducted to determine if the decontaminant exposed coupon results were statistically different compared to the control coupons.

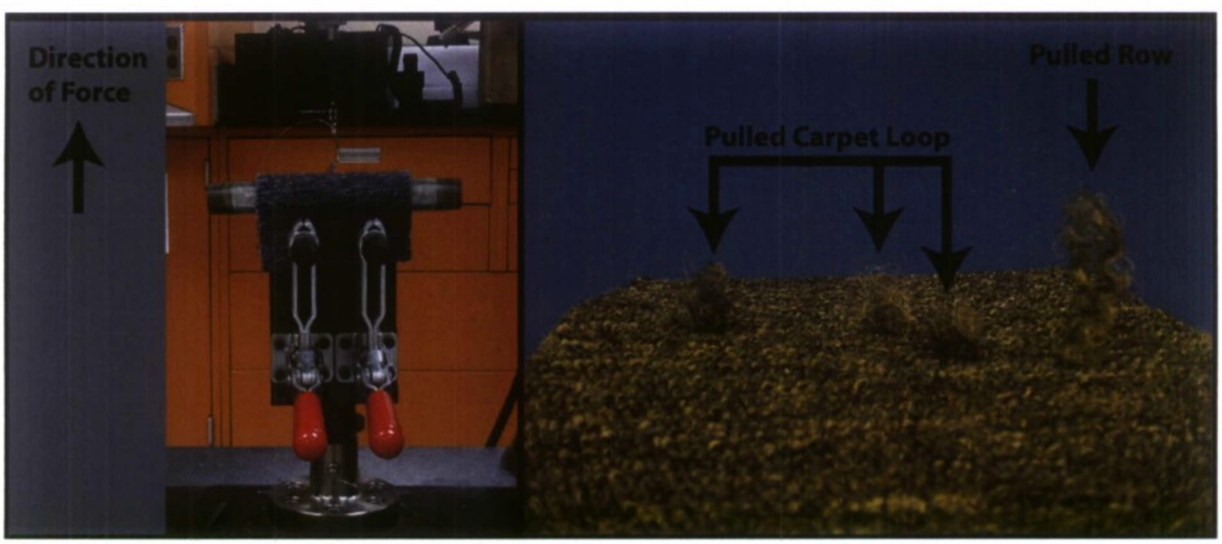


Figure 6. Representative photograph of the carpet coupon test.

8.3 Results

The coupons were stored for at least 90 days. The actual number of storage days was based on the arrival of the Instron fixture for testing. The coupons for a particular fumigation trial were studied at the same number of days. Table 6 shows the carpet tuft bind results and number of days in storage.

8.4 Discussion

A statistical analysis of the individual test results was conducted to detect potential statistical outliers (Q-Test) and determine if there are any differences between the control and exposed samples (Welch's *t* Test).

Although there was a great deal of scatter in the data (the standard deviations of thc results are between 27–35% of the mean value within the various test groups), only one of the individual tuft pulls from a single coupon (tuft one, coupon 3, RD60330) was significantly different than the others from the same carpet coupon. When comparing coupons within test groups, one coupon (coupon No. 3, RN50224, control group) was significantly different from others within the test group at the Q=0.99 confidence level. However, it was not statistically different when compared to all the control coupons. There were no statistical outliers in the half-target or full-target concentration tcsts.

The average tuft bind pull value was 16.2 ± 4.3 N for the control samples; 21.3 ± 6.8 N for the half-target concentration samples, and 17.5 ± 6.1 N for the full-target concentration samples.

Table 6. Carpet coupon test rcsults for average tuft bind.

Tuft Bind Force	0 ppm Control Sample Results (N)														
	RN50509					RN50505					RN50304				
Coupon	#1	#2	#3	#4	#5	#1	#2	#3	#4	#5	#1	#2	#3	#4	#5
Loop 1	14.9	10.8	14.3	20.9	21.6	10.8	24.3	11.7	17.0	19.8	15.1	10.3	29.8	12.1	18.1
Loop 2	8.9	17.0	15.0	19.4	16.2	16.2	20.2	11.2	17.3	18.4	20.1	23.6	15.2	13.7	18.4
Loop 3	10.0	18.2	11.8	18.0	16.4	18.1	20.9	12.0	17.7	9.9	21.9	14.7	14.6	13.6	14.7
Loop 4	16.4	13.8			16.8	21.1				16.9	10.4	15.3	20.8		
Loop 5	11.7	16.4				26.8				19.4	18.3		22.3		
Test Avg	12.4	15.2	13.7	19.5	17.7	18.6	21.8	11.6	17.4	16.9	17.1	16.0	20.6	13.1	17.1
SD	3.2	2.9	1.7	1.4	2.6	5.9	2.2	0.4	0.4	4.0	4.5	5.5	6.2	0.9	2.0
Days	158					162					151				
	RN50224														
Coupon	#1	#2	#3	#4	#5										
Loop 1	11.6	18.8	10.7	17.8	15.8										
Loop 2	13.5	7.0	9.2	12.7	15.2										
Loop 3	18.5	12.8	12.5	16.4	14.4										
Loop 4	21.3	23.6													
Loop 5	13.1	17.1													
Test Avg	15.6	15.8	10.8	15.6	15.1										
Std Dev	4.1	6.3	1.7	2.6	0.7										
Days	159														
Test Set Avg ± SD	16.2 ± 4.3														

Table 6. Carpet coupon test results for average tuft bind (continued).

Tuft Bind Force	1000 – 1250 ppm Half-Target Concentration Results (N)														
	RD60411					RD60410					RD60405				
Coupon	#1	#2	#3	#4	#5	#1	#2	#3	#4	#5	#1	#2	#3	#4	#5
Loop 1	19.4	22.1	21.0	25.6	19.9	25.3	10.9	30.1	17.1	11.6	27.0	20.5	32.3	21.7	15.2
Loop 2	19.5	26.9	21.4	29.2	22.0	24.9	15.7	20.0	20.7	16.2	19.5	41.9	25.2	17.5	14.8
Loop 3	12.3	23.4	25.9	31.1	19.3	20.0	23.9	19.5	14.6	23.3	13.4	22.7	11.9	20.7	28.6
Loop 4	14.4						11.5	23.8	15.5	26.5	34.5	13.5	26.0		19.9
Loop 5	22.3						12.1	21.2		11.7	23.1	15.3	42.7		18.8
Test Avg	17.6	24.2	22.8	28.7	20.4	23.4	14.9	22.9	17.0	17.9	23.5	22.8	27.7	20.0	19.5
Std Dev	4.1	2.5	2.7	2.8	1.4	2.9	5.4	4.4	2.7	6.8	7.9	11.3	11.3	2.2	5.6
Days	163					164					169				
Test Set Avg ± SD	21.3 ± 6.8														

Tuft Bind Force	2000 – 2500 ppm Full-Target Concentration Results (N)														
	RD60330					RD60406					RD60404				
Coupon	#1	#2	#3	#4	#5	#1	#2	#3	#4	#5	#1	#2	#3	#4	#5
Loop 1	26.8	16.3	15.8	17.3	18.9	21.9	15.7	24.6	18.8	14.2	6.2	13.4	23.1	23.2	7.9
Loop 2	24.9	26.3	20.8	15.6	14.1	19.7	22.8	14.4	13.1	17.9	3.5	13.0	14.0	15.1	4.9
Loop 3	26.2	15.1	20.8	15.0	22.8	15.8	21.4	21.9	21.5	15.3	4.7	13.1	15.4	16.4	6.6
Loop 4		20.9			13.3	21.3	21.6	18.8	21.5		28.2		13.2	27.1	6.5
Loop 5		15.4			24.2	16.3	17.0	32.3	18.0		15.4		18.6	22.3	9.5
Test Avg	25.9	18.8	19.1	16.0	18.7	19.0	19.7	22.4	18.6	15.8	11.6	13.2	16.9	20.8	7.1
Std Dev	1.0	4.8	2.9	1.2	5.0	2.8	3.1	6.7	3.4	1.9	10.4	0.2	4.1	5.0	1.7
Days	175					168					170				
Test Set Avg ± SD	17.5 ± 6.1														

Notes: The blank cells are samples that were not required to be analyzed, due to meeting the test method sampling criteria of ±15%. The cells highlighted in orange were determined to be outliers according to the Q-Test at the 99% confidence interval within their test sets, but not within the test group (four control groups, three target concentration groups); therefore, the values were retained.

A Welch's t Test analysis was conducted on the samples to determine if there were statistical differences between the control, half-target, and full-target concentration samples. The half-target concentration results were determined to be significantly different statistically from the control and target samples at the 95% confidence level, an unexpected result due to the large standard deviations of the three sets of data and their respective overlaps. The control and target concentration samples were not found to be significantly different at the 95% confidence level. The lack of difference for the target concentration samples and control samples indicated that the difference between the half-target data was not likely due entirely to ClO_2.

A minor increase for the average tuft bind results with exposure to ClO_2 was apparent, but the trend was smaller than the standard deviations of the individual test results, so it was not clear whether it was an experimental artifact or a real trend.

These test methods show that exposure to ClO_2 may have a statistically significant effect on the tuft bind pull tests of carpet; further study would be required to define the nature and magnitude of the effect.

9. EVALUATION OF CONCRETE CINDER BLOCK

9.1 Introduction

The effects of ClO_2 on the physical integrity of concrete cinder block coupons were investigated using the compression test as described in ASTM Test C140-03 "Standard Test Methods for Sampling and Testing Concrete Masonry Units and Related Units." The ASTM test was used to determine the integrity of the concrete cinder block coupons exposed to vaporous decontaminant compared to unexposed (control) concrete cinder block coupons.

9.2 Sample Preparation and Testing

The concrete cinder block samples were removed from storage, visually inspected, and measured. The coupons from chamber positions 1, 4, and 7 were selected for testing; the coupons were selected to obtain representation thoughout the test chamber. The samples were brought to equilibrium in an environmental range of 16–32 °C and less than 80% RH for 48 h prior to testing. The testing was conducted in accordance with the ASTM test method C140-03. The Instron fixture for the concrete cinder block test was installed prior to testing. The Instron universal testing machine operation and calibration verification was conducted by suspending a certified weight from the fixture and recording the weight. A photograph of a test sample loaded into the Instron test fixture is shown in Figure 7. Three sets of three coupons were tested for each concentration (0 ppm, full-target and half-target). The load required to rupture the coupons was measured in N. No precision or bias requirements were established for this test method. The results of control coupons were compared to decontaminant exposed samples. A statistical analysis of the data was conducted to determine if the decontaminant exposed coupon results were statistically different compared to the control coupons.

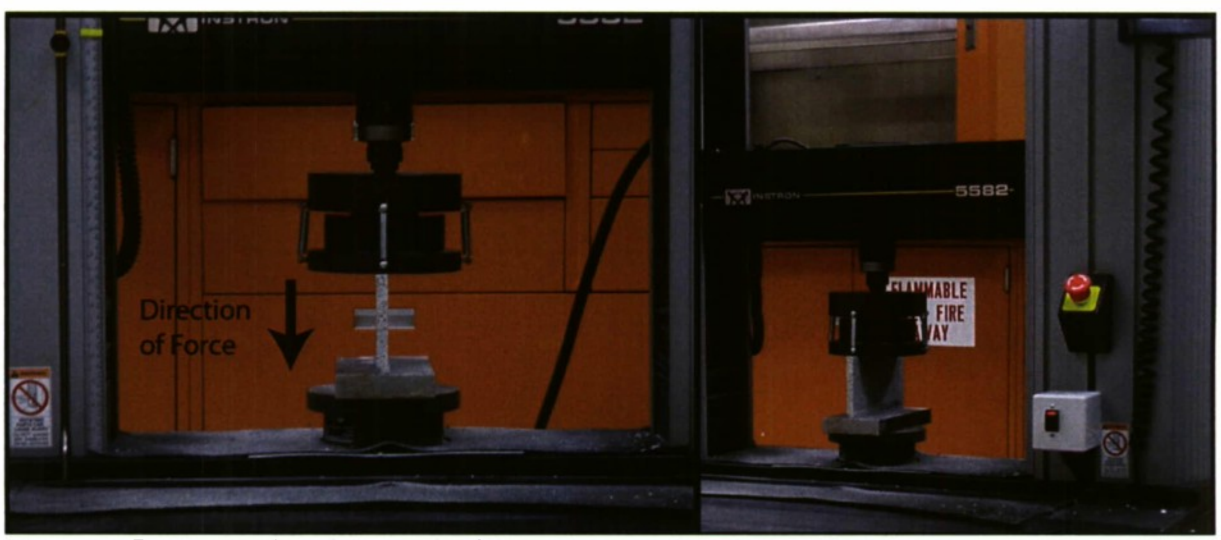


Figure 7. Representative photograph of the concrete cinder block coupon test.

9.3 Results

The coupons were stored for at least 90 days. The actual number of storage days was based on the arrival of the Instron fixture for testing. The coupons for a particular fumigation trial were studied at the same number of days. Figure 8 shows a photograph of a representative concrete cinder block sample before and after testing. The coloring difference between the pictures is a result of the room lighting. Both samples were taken on the same blue color mat. The load required to crush the concrete cinder block coupons, the coupon gross area compressive strength results, and number of days in storage values are provided in Table 7. The concrete cinder block was a heterogeneous material sample to sample. The break patterns varied from sample to sample; a photograph of each sample is provided in Appendix C.

9.4 Discussion

A statistical analysis of the individual test results was conducted to detect potential statistical outliers (Q-Test) and determine if there is a difference between the control and exposed samples (Welch's *t* Test). Within individual test runs for maximum load and gross area compressive strength, there were no statistical outliers.

The values for the maximum load tests for concrete cinder blocks were 3239 ± 729 kgf for the control samples, 3617 ± 776 kgf for the half-target concentration samples, and 3512 ± 898 kgf for the full-target concentration samples. The values calculated for the gross compressive strength of the cinder block samples were 1.6 ± 0.4 kgf/mm^2 for the control samples, 1.7 ± 0.4 kgf/mm^2 for the half-target samples, and 1.7 ± 0.5 kgf/mm^2 for the full-target samples.

Comparing individual test set averages and gross area compressive strength within test groups, there were no statistical outliers.

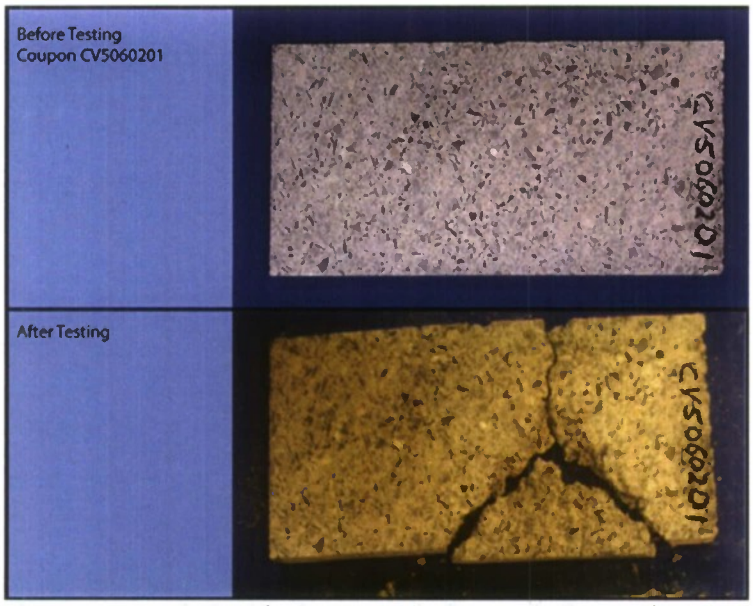


Figure 8. Representative concrete cinder block coupon before and after testing.

Table 7. ClO_2 cinder block coupon test results.

Maximum Load	0 ppm Control Samples Results (kgf)			1000 – 1250 ppm Half-Target Conc. Results (kgf)			2000 – 2500 ppm Full-Target Conc. Results (kgf)		
	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon Set	CN50510	CN50524	CN51027	CD51129	CD51201	CD51205	CD51011	CD51012	CD51013
Coupon 1	3760	3252	4880	4243	3619	3091	5372	2959	3558
Coupon 2	3112	2711	3011	3001	4458	4107	2871	2376	3839
Coupon 3	2554	2557	3310	3074	2366	4596	3818	2790	4027
Test Avg	3142	2840	3734	3439	3481	3931	4020	2708	3808
Std Dev	603	365	1004	697	1053	768	1262	300	236
Test Set Avg ± Std Dev	3239 ± 729			3617 ± 776			3512 ± 898		
Gross Area Compressive Strength	**Control Samples Results (kgf/mm^2)**			**Half-Target Concentration Results (kgf/mm^2)**			**Full-Target Concentration Results (kgf/mm^2)**		
	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon Set	CN50510	CN50524	CN51027	CD51129	CD51201	CD51205	CD51011	CD51012	CD51013
Coupon 1	1.9	1.5	2.3	2.2	1.7	1.6	2.8	1.4	1.8
Coupon 2	1.5	1.4	1.6	1.3	2.1	1.8	1.4	1.1	2.1
Coupon 3	1.1	1.2	1.7	1.5	1.2	2.2	2.0	1.3	1.8
Test Avg	1.5	1.4	1.9	1.7	1.7	1.9	2.0	1.3	1.9
Std Dev	0.4	0.2	0.4	0.5	0.5	0.3	0.7	0.1	0.2
Test Set Avg ± Std Dev	1.6 ± 0.4			1.7 ± 0.4			1.7 ± 0.5		
Number of Days in Storage	**Control Samples (Days)**			**Half-Target Concentration (Days)**			**Full-Target Concentration (Days)**		
	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon Set	CN50510	CN50524	CN51027	CD51129	CD51201	CD51205	CD51011	CD51012	CD51013
Coupon 1	126	127	219	128	126	211	162	211	160
Coupon 2	126	127	219	128	126	211	162	211	160
Coupon 3	126	127	219	128	126	211	162	211	160
Test Set Avg ± Std Dev	157 ± 46			155 ± 42			178 ± 25		

When the averages for the exposed and non-exposed coupons were compared, no statistical differences were found among them. The Welch's *t* Test evaluation of the data indicated that there were no statistically significant differences among the means of the exposed and control samples at the 95% confidence level. These test methods showed that exposure to ClO_2 has no statistically significant effect on the maximum load or the gross area compressive strength of the cinder blocks tested.

10. EVALUATION OF WOOD

10.1 Introduction

The effects of ClO_2 on the physical integrity of wood were investigated using the bending edge-wise test as described in ASTM Test D4761-02a "Standard Test Methods for Mechanical Properties of Lumber and Wood-Base Structural Material," Sections 6–11. The ASTM test was used to determine the integrity of the wood coupons exposed to vaporous decontaminant compared to unexposed (control) wood coupons.

10.2 Sample Preparation

The wood samples were removed from storage, visually inspected, and measured. The coupons from chamber positions 1, 4, 7, 10, and 14 were selected for testing; the coupons were selected to obtain representation thoughout the test chamber. The samples were brought to moisture equilibrium such that the weight of the sample did not change by more than 0.2% on successive weighings at a minimum interval of 2 h. The sample preparation was conducted within a temperature range of 15–25 °C and a RH of 48–75%. The testing was conducted in accordance with the ASTM test method D4761-02a. The Instron fixture for the wood test was installed prior to testing. The Instron universal testing machine operation and calibration verification was conducted by suspending a certified weight from the fixture and recording the weight. Three sets of five coupons were tested for each concentration (target and half-target) and four sets were tested for the controls (0 ppm). The load required to rupture to the wood coupons was measured in N. No precision or bias requirements were established for this test method. The results of control coupons were compared to decontaminant exposed samples. A statistical analysis of the data was conducted to determine if the decontaminant exposed coupon results were statistically different compared to the control coupons. A photograph of a wood sample loaded into the Instron is provided in Figure 9.

10.3 Results

The coupons were stored for at least 90 days. The actual number of storage days was based on the arrival of the Instron fixtures for testing. The coupons for a particular fumigation trial were studied at the same number of days. A photograph of a representative wood sample before and after testing is provided in Figure 10. The wood coupon results for the required load and time to break, moisture content, and number of days in storage are provided in Table 8. The wood samples vary slightly in knot and grain pattern from sample to sample. The break patterns varied from sample to sample; a photograph of each sample is provided in Appendix D.

10.4 Discussion

Of the 50 coupons tested to destruction in this portion of the program, no coupons were eliminated as statistical outliers from within their individual test sets or test groups (control, half-target concentration, or full-target concentration samples) at the Q=0.99 level of confidence.

When considering the data from the test groups of coupons, the average maximum load values for the half-target ClO_2 exposed coupons increases slightly (approximately 1%) over the value for the control set, while the maximum load drops by 17% for the full concentration coupons, with relative standard deviations on the order of 21–28%. The average maximum force value for the control samples was determined to be 4283 ± 1009 N. The half-concentration samples were determined to have had an average maximum force value of 4342 ± 1201 N (an increase of 1.4%); whereas, the full-concentration samples had an average maximum force value of 3558 ± 731 N (a decrease of 17.4% from the control group). The maximum force for the full-concentration exposed coupons statistically was significantly different from the controls and the half-concentration coupons at a 95% confidence level using the Welch's t Test results.

Average time to break values for the control coupons was 4.1 ± 0.9 s half-concentration coupons was 4.0 ± 0.7 seconds (a 2% decrease), and the full-concentration coupons was 3.5 ± 0.7 seconds (a 15% decrease). The time to break values for the exposed coupons showed a slight downward trend. The Welch's t Test was again used to compare the time to break values of the different groups of coupons. At the 95% confidence level, the full concentration coupons were found to be significantly different from the control and the half-target coupons.

The average change in moisture content for the control samples after storage was -2.32 ± 5.95%. For the half-concentration coupons, the average change in moisture content was +0.01 ± 0.11%, and for the full-concentration coupons the average change in moisture content was -1.24 ± 0.23%. The changes in moisture content were not suggested to be significantly different statistically at the 95% confidence level using the Welch's t Test.

The results suggest that fumigation at the full-target conditions used in this study may impact the force required to break the structural wood, in accordance with the ASTM test method used. At the higher fumigant concentration, the wood samples required less force and time to break than either the controls or half-target samples.

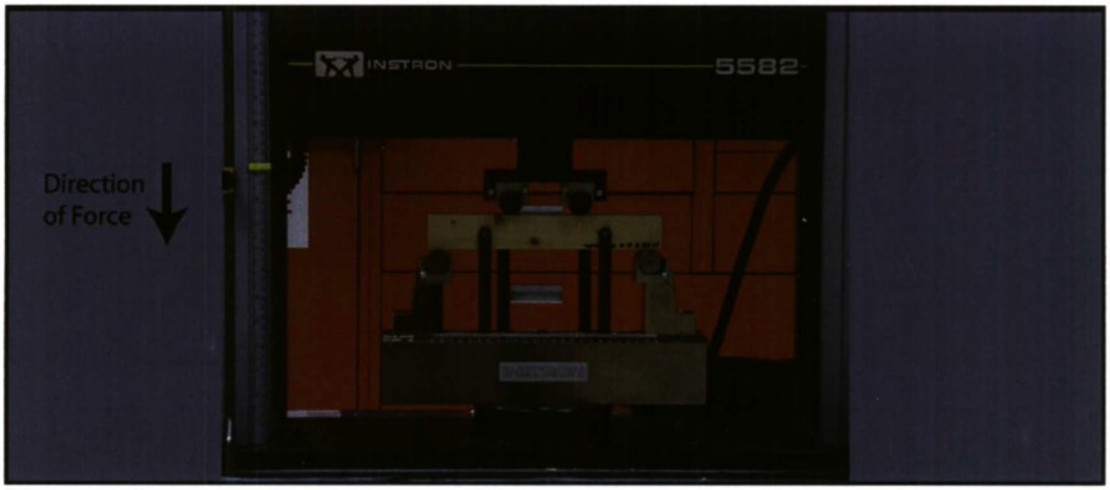


Figure 9. Representative photograph of the wood coupon test.

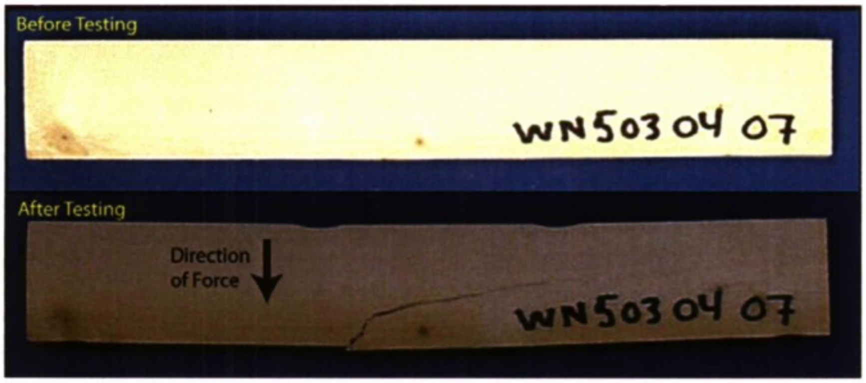


Figure 10. Representative wood coupon before and after testing.

Table 8. ClO_2 coupon test results for wood.

Maximum Force	0 ppm Control Samples Results (N)				1000 – 1250 ppm Half-Target Concentration Results (N)			2000 – 2500 ppm Full-Target Concentration Results (N)		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon Set	WN5 0307	WN5 0317	WN5 0502	WN5 0504	WD6 0213	WD6 0207	WD6 0209	WD5 1206	WD5 1207	WD5 1208
Coupon 1	4945	3903	4516	4600	2360	4163	4712	4683	1906	3037
Coupon 2	2433	3546	5582	3248	3766	4714	4289	4514	3027	3860
Coupon 3	5130	2574	4031	5233	3312	3994	7141	3699	3516	3131
Coupon 4	3592	3494	6370	5170	3702	3651	3779	3741	4427	3095
Coupon 5	4825	4446	3475	4545	4015	4983	6553	4193	2995	3543
Test Avg	4185	3593	4795	4559	3431	4301	5295	4166	3174	3333
Std Dev	1151	684	1173	798	650	541	1470	444	915	356
Test Set Avg ± Std Dev	4283 ± 1009				4342 ± 1201			3558 ± 731		
Time to Break	Control Samples Results (min)				Half-Target Concentration Results (min)			Full-Target Concentration Results (min)		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon Set	WN5 0307	WN5 0317	WN5 0502	WN5 0504	WD6 0213	WD6 0207	WD6 0209	WD5 1206	WD5 1207	WD5 1208
Coupon 1	4.9	3.9	4.3	4.6	2.4	4.1	4.7	4.7	1.9	3.0
Coupon 2	2.4	3.6	5.6	3.2	3.7	4.7	4.3	4.5	3.0	3.9
Coupon 3	5.1	2.6	4.0	5.2	3.3	4.0	4.9	3.7	3.5	3.1
Coupon 4	3.6	3.5	6.1	4.6	3.7	3.6	3.8	3.7	4.3	3.1
Coupon 5	4.8	4.4	3.4	4.4	4.0	5.0	4.5	4.2	2.9	3.5
Test Avg	4.2	3.6	4.7	4.4	3.4	4.3	4.4	4.2	3.1	3.3
Std Dev	1.1	0.7	1.1	0.7	0.6	0.5	0.4	0.4	0.9	0.4
Test Set Avg ± Std Dev	4.2 ± 1.0				4.0 ± 0.7			3.5 ± 0.7		
Moisture Content	Control Samples %				Half-Target Concentration %			Full-Target Concentration %		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon Set	WN5 0307	WN5 0317	WN5 0502	WN5 0504	WD6 0213	WD6 0207	WD6 0209	WD5 1206	WD5 1207	WD5 1208
Coupon 1		0.12	-0.12	-0.40	-0.18	0.03	0.07	-1.00	-0.93	-0.84
Coupon 2		0.20	-0.17	-0.33	-0.14	0.11	0.00	-1.27	-1.31	-1.27
Coupon 3		0.17	-0.14	-16.61	-0.03	0.13	0.02	-1.29	-1.39	-1.30
Coupon 4		0.12	-0.13	-17.29	-0.12	0.14	0.00	-1.34	-1.39	-1.67
Coupon 5		0.17	-0.13	-0.24	-0.12	0.20	0.00	-1.53	-1.15	-1.00
Test Avg		0.15	-0.14	-6.98	-0.12	0.13	0.02	-1.29	-1.24	-1.21
Std Dev		0.03	0.02	9.11	0.05	0.06	0.03	0.19	0.20	0.32
Test Set Avg ± Std Dev	-2.32 ± 5.95				0.01 ± 0.11			-1.24 ± 0.23		
Number of Days in Storage	Control Samples Days				Half-Target Concentration Days			Full-Target Concentration Days		
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 1	Test 2	Test 3
Coupon Set	WN5 0307	WN5 0317	WN5 0502	WN5 0504	WD6 0213	WD6 0207	WD6 0209	WD5 1206	WD5 1207	WD5 1208
Days	135	125	182	372	220	227	225	149	148	147
Test Set Avg ± Std Dev	204 ± 102				224 ± 3			148 ± 1		

Note: Gray cells indicate that there was no data collected.

11. EVALUATION OF ELECTRICAL CIRCUIT BREAKERS

11.1 Introduction

The impact of fumigant and humidity on the performance of electrical circuit breakers post-treatment was also investigated in this study. This investigation involved circuit breakers prepared as baseline, test, and control. Baseline circuit breakers were the as-purchased circuit breakers. The test circuit breakers were prepared in the exposure chambers using fumigant. The control circuit breakers were prepared in the exposure chambers using a temperature and RH profile similar to that of the test breakers.

11.2 Sample Preparation

The single pole, 20 amp rated circuit breakers were purchased from the Home Depot (model HOM120). All of the circuit breakers were installed in the testing stations to confirm they were operational before exposure testing. All of the circuit breakers were removed from the stations, numbered, and COC initiated. The baseline circuit breakers were put aside until needed. The test and control exposure testing was discussed in Section 4. Each run used seven circuit breakers. After a test or control circuit breaker set was prepared in the exposure chamber, the breakers were removed from the exposure chamber and visually inspected.

11.3 Circuit Breaker Testing Stations

After visual inspection, the breakers were installed in the testing station and observed for 90 days under load. A photograph of the testing station is shown in Figure 11. The testing station is an electrical box containing eight spaces, 16 circuits, 100 amp max from square D (Home Depot No. 577-340). The circuit breaker box was wired with 12 gauge, 20 amp wire into the 120 V outlet. Each circuit breaker was wired in series with an electrical lamp (s513e) with an outlet box (s110e) manufactured by Thomas & Bretts (Home Depot No. c214477 and b214426, respectively). Each lamp contained a Phillips 40 watt light bulb (Home Depot No. a356140). The test or control circuit breakers were installed into slots 1 though 7 and the baseline circuit breaker was installed in slot 8 (Figure 11, upper left picture). The room temperature and RH were monitored daily.

11.4 Results

The circuit breakers were exposed to fumigant and visually inspected after removal from the exposure chamber. No visual damage was observed on any of the circuit breakers used in this program following fumigation. The circuit breakers were then installed into the testing stations for 90 days. The stations were observed on each work day and light bulbs replaced as needed. No breakers failed during the 90 day storage under load. Following the 90 day storage, the breakers were tested using current-time measurements at 150% (30 amp) and 300% (60 amp) of the breakers' rated value. Tests were performed using an AVO/multi-amp MS-2, available from Advanced Test Equipment Rentals. The test results were provided in Table 9. The circuit breaker data were statistically analyzed to determine if the breakers were compromised after exposure to decontaminant by comparing the test results obtained with fumigant-exposed circuit breakers to those obtained with control coupons (not exposed to fumigant). Each breaker station contained one control breaker that had not been exposed in the chamber.

The measurement for the analysis was the time for the circuit breaker to open (Time to Open) when experiencing a current above its rated value. A circuit breaker that trips too quickly will protect personnel and equipment, but can represent a significant loss of time and productivity, as well as a major source of frustration for all involved. A circuit breaker that takes too long to trip could result in a heat buildup and possibly a fire, resulting in a failure to protect equipment, users, and property.

A statistical analysis of the individual test results was conducted to detect potential statistical outliers (Q-Test) and determine if there were any differences between the control and exposed samples (Welch's *t* Test) in the tests of circuit breakers exposed to ClO_2.

The differing test groups were first checked to determine if there were any statistical outliers using the Q-Test. There were two statistical outliers found in the data at the Q=0.99 level of confidence: circuit breaker BD5090601 from the 6 h control tested at the 30 amp challenge and circuit breaker BN5022804 from the 12 h exposed group tested with the 60 amp challenge. These data points are highlighted in orange in Table 9 and were discarded; the remainder of the statistical analysis was conducted without them.

Table 10 summarizes the data for the average and standard deviation for the various test groups. The Welch's *t* Test was used with a 95% confidence level to determine if the changes in the Time to Open were significantly different among the different treatment (control, 1000 ppm, or 2000 ppm) and analysis challenge (30 amp or 60 amp) groupings. At the 60 amp challenge, the slight decreases in the Time to Open for the circuit breakers exposed to the ClO_2 fumigation conditions compared to their respective controls were not statistically significant. In addition, no statistically significant difference was observed due to exposure to the control conditions for 6 compared to 12 h, nor as a function of fumigation conditions.

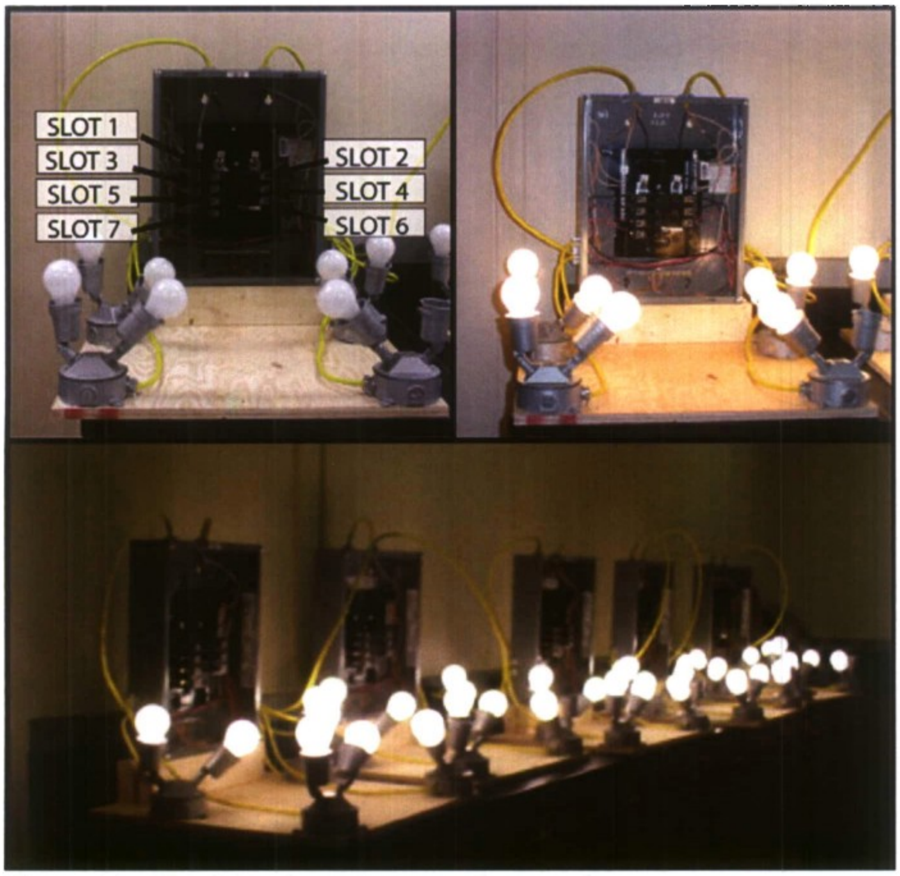


Figure 11. Circuit breaker test station photograph.

Table 9. ClO_2 circuit breaker test results.

6 H ClO_2 Box Test	60 Amp Test Time (sec)	30 Amp Test Time (sec)	6 H ClO_2 Control	60 Amp Test Time (sec)	30 Amp Test Time (sec)
BD5090601	4.65	223.70	BN5022501	5.37	43.21
BD5090602	5.16	82.72	BN5022502	5.81	57.52
BD5090603	4.53	90.65	BN5022503	5.47	55.42
BD5090604	6.59	81.82	BN5022504	5.75	61.22
BD5090605	5.05	115.87	BN5022505	4.85	48.62
BD5090606	3.45	64.91	BN5022506	5.52	50.31
BD5090607		64.87	BN5022507	5.37	48.45
			BN5022506 Retest		55.48
Control	6.08	62.14	BN50225NA	4.97	41.47
Test Average	4.91	103.51	Test Average	5.45	52.53
Standard Deviation	1.02	55.75	Standard Deviation	0.32	5.87
12 H ClO_2 Box Test	**60 Amp Test Time (sec)**	**30 Amp Test Time (sec)**	**12 H ClO_2 Control**	**60 Amp Test Time (sec)**	**30 Amp Test Time (sec)**
BD5091501	5.34	61.25	BN5022801	5.54	57.50
BD5091502	4.40	90.23	BN5022802	5.59	51.08
BD5091503	5.14	68.25	BN5022803	6.41	55.60
BD5091504	6.11	60.90	BN5022804	2.99	53.29
BD5091505	3.05	53.95	BN5022805	6.00	51.94
BD5091506	4.81	64.55	BN5022806	5.41	68.59
BD5091507	3.64	76.24	BN5022807	5.98	48.79
Control	3.28	60.99	BN50228NA	5.68	63.89
Test Average	4.64	67.94	Test Average	5.68	63.89
Standard Deviation	1.04	12.04	Standard Deviation	1.12	6.55

Table 10. Summary of time to open data.

Exposure	30 Amp Challenge Time to Open (sec)	60 Amp Challenge Time to Open (sec)
6 H Control	52.53 ± 5.87	5.45 ± 0.32
6 H @ 2000 ppm ClO_2	83.47 ± 18.95	4.91 ± 1.02
12 H Control	55.26 ± 6.55	5.82 ± 0.38
12 H @ 1000 ppm ClO_2	67.94 ± 12.04	4.64 ± 1.04

However, at the 30 amp challenge, the decrease in the Time to Open for the circuit breakers exposed to 2000 ppm of ClO_2 for 6 h was significantly different statistically from the corresponding 6 h control. At the lower concentration and longer fumigation time, the observed slight increase in time to open compared to the 12 h control was not a statistically significant difference. As with the 60 amp challenge, no statistically significant difference was observed due to exposure to the control conditions for 6 compared to 12 h. In addition, the observed difference in the time to open for the 6 h versus 12 h ClO_2-exposed circuit breakers was also not statistically significant.

The results are somewhat conflicting, i.e., no statistically significant difference between the 6 h and 12 h ClO_2 exposure for the 60 amp challenge. However, it appeared that exposure to the higher concentration of ClO_2 may have an effect on the performance of a circuit breaker as determined from the Time to Open testing based on the comparison to the control. The results suggested that exposure to lower ClO_2 concentrations for longer times, rather than high concentrations for shorter times, may have less deleterious effects on the circuit breakers. No specification was found to determine if the observed effect at the 2000 ppm ClO_2 fumigation condition was within the device failure criteria.

12. ClO_2 FATE ON METALS

12.1 Introduction

This study characterized the interaction of ClO_2 with aluminum, copper, and steel; materials common in electrical systems. Residual chloride byproducts on the metals following treatment with ClO_2 were analyzed using ion chomatography. Analyses for other possible reaction products, such as metal hydroxides and oxides, were attempted. The objective of this study was to identify the anionic chlorine species and concentrations formed on aluminum, steel, and copper after exposure to ClO_2 at 1000 and 2000 ppm (parts per million volume) for 12 and 6 h, respectively.

12.2 Test Procedure

Aluminum (0.5 in. W x 0.5 in. L), copper (0.5 in. D x 0.5 in. L), and steel EMT conduit (0.5 in. D x 0.5 in.) coupons were exposed to ClO_2 to determine the fate of the decontaminant on the metals. Coupons cut from metals purchased from Home Depot were exposed to ClO_2 concentrations of 1000 and 2000 ppm for 12 and 6 h, respectively, to give a total CT of 12,000 ppm-h for each experiment. The metals were exposed at 25 °C, 75–90% RH; control sets were exposed under the same conditions at 0 ppm ClO_2. Fourteen coupons of each metal in small plastic containers were placed on the inside bottom of a PlasLabs model 830-ABC glovebox (PlasLabs, Inc., 401 E. North Street, Lansing MI 4890) during each exposure. The coupons were removed from the glovebox after exposure and stored for 90 days in the plastic containers. After storage, each of the 14 coupons was placed in a 2 ounce wide mouth glass jar with cap. Chloride, chlorite, chlorate, perchlorate, and other anions were removed from each sample by extraction in water (10 mL) with rocking. The extracts were then filtered though a 0.22 µm syringe filter and injected directly into the chomatograph to determine retention time and detector response for each analyte present in the sample. De-ionized distilled water was used to dilute samples, when appropriate. The anions were then identified and quantified by comparing with standard solutions.

12.3 Sample Analyses

Sodium carbonate (analytical grade) and HPLC grade methanol used in preparing the mobile phase was purchased from Fisher Scientific (Fair Lawn, NJ). The chloride, chlorate, and perchlorate anion standards were obtained from SCP Scientific, Champlain, NY. The chlorite anion standard was obtained from HPS Science, Charleston, SC. The analyses were carried out using an ion chomatograph with a Millennium32 Data Workstation equipped with a Rheodyne 77251i Injector, a

Model 510 Pump, and a Model 432 Conductivity Detector (Waters Corporation, Milford, MA). Conductivity suppression was carried out using an ERIS 1000HP Autosuppressor (Alltech Corporation, Deerfield, IL).

Analyses for chloride, chlorite, and chlorate in the extracts were performed under thc following conditions: column, ION-PAC AS9HC (Dionex Corporation, Sunnyvale, CA); mobile phase, 9 mM Na_2CO_3; flow rate, 1.0 mL/min; injection volume, 20 µL; and detection, suppressed conductivity (1 SFS). Standard solutions of each anion were injected onto the column and rctention times of each analyte determined. Calibration curves were obtained by injecting known concentrations (100, 40, 10, 1, and 0.4 µg/mL) of each anion in de-ionized water into the chomatograph in duplicate and measuring the conductivity response obtained.

Analysis of the extracts for perchlorate were performed under the following conditions: column, ION-PAC AS 14 (4 mm) (Dionex Corporation, Sunnyvale, CA); mobile phase, 9 mM Na_2CO_3 in 40% methanol; flow rate, 1.0 mL/min., injection volume, 20 µL; detection, suppressed conductivity (1 SFS). Standard solutions of perchlorate were injected onto the column and a retention time was determined. A calibration curve of perchlorate ion was obtained by injecting a known concentration (100, 40, 10, 4, 1, 0.4 µg/mL) of the anion in de-ionized water into the chomatograph in duplicate and measuring the conductivity responsc obtained.

The detector response versus concentration for all of the spccies was determined to be linear. A typical regression line for each was determined using the least square method. The regression curve and linear correlation coefficient (R2) for each target analyte were determined to be as follows: chloride, $y = 118486x - 136041$ (R2 = 0.9987); chlorite, $y = 42201X - 53598$ (R2 = 0.9979); chlorate, $y = 49100X - 43882$ (R2 = 0.9982); and perchlorate, $y = 151113X - 135662$ (R2 = 0.9996). The reproducibility was determined to bc within ±5% of the mean.

Analyte standards at the 10 µg/mL concentration levcl were injected before and after a daily analytical run with reproducibility having to be within ±10% of mean. The unknown concentration of each species in the extracts was determined by correlating the detector response to the response versus concentration curve (standard curve) obtained for each target analyte. The detector response was substituted into the appropriate regression equation and the corresponding concentration was calculated.

12.4 Results

A summary of the ClO_2 reactions on aluminum, copper, and iron with the corresponding products and concentrations arc provided in Table 11. A photographic comparison of copper tubing not exposed and exposed to ClO_2 is provided in Figures 12 and 13, respectively. A layer of patina is evident on the tubing exposed to ClO_2 (Figure 13). Ion chomatography analyses of the extraction solvents from the aluminum controls detected chloride ions (1.59 µg), chlorite ions (7.96 µg), and chlorate ions (11.83 µg). Analyses of iron and copper controls identified only chloride ions, 14.58 and 31.78 µg, respectively.

The reaction of ClO_2 (6 h at approximately 2000 ppm) with aluminum yielded four metal salts: $AlCl_3$, $Al(ClO_2)_3$, $Al(ClO_3)_3$, and $Al(ClO_4)_3$. Decomposition at the lower concentration of ClO_2 (12 h at approximately 1000 ppm) produced only chloride, chlorite, and chlorate ions. The amounts of anions, except for chloride, were grcatcr in the reaction with the highcr concentration of ClO_2.

The reaction of ClO_2 with iron gave three metal salts: $FeCl_3$, $Fc(ClO_3)_3$, and $Fe(ClO_4)_3$. Decomposition in 2000 ppmv (6 h) ClO_2 gave about 39% more chloride than at 1000 ppmv (12 h); however, the amounts of the other anions were similar between the two reactions.

Decomposition of ClO_2 on copper (12 h at 1000 ppm ClO_2) yielded $CuCl_2$ (most abundant), $Cu(ClO_3)_2$, and $Cu(ClO_4)_2$ complexes. ClO_2 (6 h at 2000 ppmv) produced predominantly chloride and a relatively lower concentration of chlorate ions.

Table 11. Residual anions on metal coupons after exposure to ClO_2.

Sample (Treatment time and concentration)	Amount Analyte Present (µg)			
	Chloride (Cl^-)	Chlorite (ClO_2^-)	Chlorate (ClO_3^-)	Perchlorate (ClO_4^-)
Aluminum (control)	1.59	7.96	11.83	BDL[a]
Aluminum (6 h ~ 2000 ppmv)	933.5	31.48	133.9	45.48
Aluminum (12 h ~ 1000 ppmv)	1,314	19.72	15.76	BDL[a]
Iron (control)	14.58	BDL[a]	BDL[a]	BDL[a]
Iron (6 h ~ 2000 ppmv)	22,383	BDL[a]	26.52	39.46
Iron (12 h ~ 1000 ppmv)	16,062	BDL[a]	21.79	37.60
Copper (control)	31.78	BDL[a]	BDL[a]	BDL[a]
Copper (6 h ~ 2000 ppmv)	942.5	BDL[a]	40.59	BDL[a]
Copper (12 h ~ 1000 ppmv)	1,630	BDL[a]	76.78	37.46

Note: [a]Below the detection limit (less than 0.4 µg)

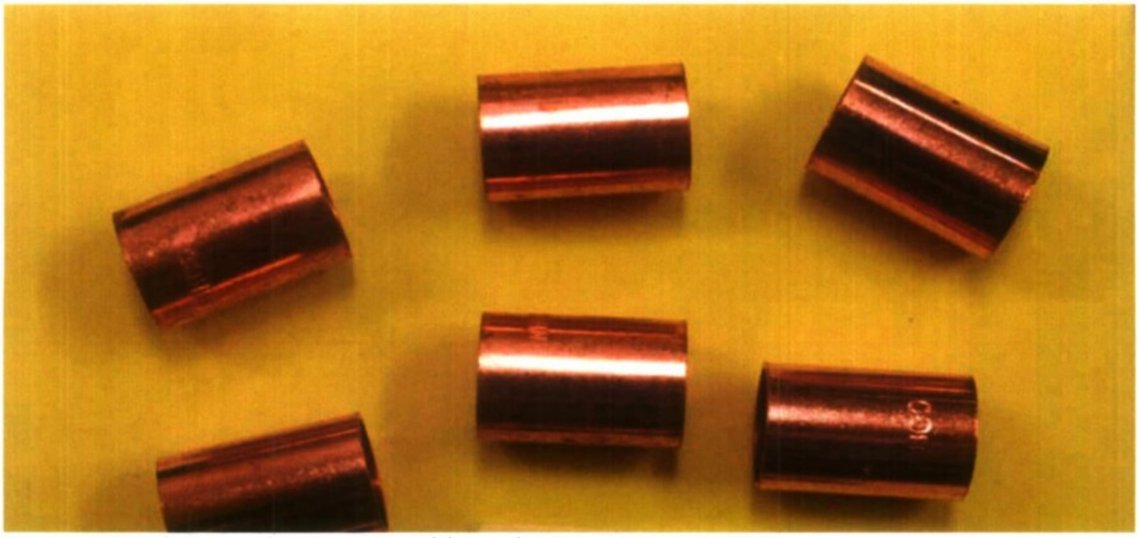


Figure 12. Photograph of copper tubing prior to exposure to ClO_2.

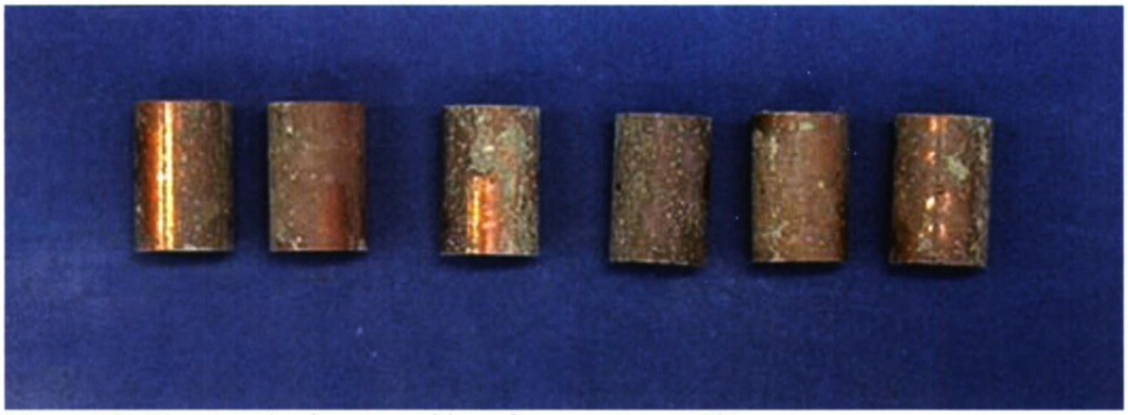


Figure 13. Photograph of copper tubing after exposure to ClO_2.

13. QUALITY ASSURANCE FINDINGS

Three technical audits of the Instron destructive testing process on ClO_2-fumigated coupons were conducted over the course of the program. The first technical audit, conducted 6 June 2005, covered steel coupons from a control run in the ClO_2 chamber. A second technical audit was conducted on 21 September 2006, involving carpet samples fumigated with ClO_2. A third technical audit was conducted on 22 September 2006, involving wood and gypsum wallboard samples fumigated with ClO_2. All operations were conducted in accordance with the applicable SOPs and IOPs. Data quality audits were conducted on 8 of the 63 ClO_2 material compatibility tests (13%). All were found to be acceptable, in accordance with the QAPP.

LITERATURE CITED

1. Quality Assurance Project Plan and Work Plan for Deposition Velocity Studies: Materials Sorption of Vaporized Hydrogen Peroxide or Chlorine Dioxide; Version 3.0; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Grounds, MD, 2004.

2. *Quality Management Plan (QMP) for the National Homeland Security Research Center (NHSRC) Office of Research and Development (ORD).* U.S. Environmental Protection Agency: Washington, DC, 2003.

3. *Quality Management Plan for Environmental Programs;* U.S. Army Edgewood Chemical Biological Center, US Army Research, Development and Engineering Command, Aberdeen Proving Ground, MD 2003.

4. *EPA Guidance for Data Quality Assessment, Practical Methods for Data Analysis*; EPA QA/G-9; U.S. Environmental Protection Agency: Washington, DC, 2000.

5. *EPA Requirements for Quality Assurance Project Plans*; EPA QA/R-5; U.S. Environmental Protection Agency: Washington, DC, 2001.

6. *EPA Guidance for Quality Assurance Project Plans*; EPA QA/G-5; U.S. Environmental Protection Agency: Washington, DC, 2002.

7. *EPA Guidance on Environmental Data Verification and Data Validation*; EPA QA/G-8; U.S. Environmental Protection Agency: Washington, DC, 2002.

8. Quality Assurance Project Plan and Work Plan for Effects of Vaporized Decontamination Systems on Selected Building Interior Materials; Version 3.0; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Grounds, MD, 2004.

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ACRONYMS

ASTM	American Society for Testing and Materials
CB	Chemical and Biological
ClO_2	Chlorine Dioxide
COC	Chain of Custody
CT	Concentration Time
ECBC	Edgewood Chemical Biological Center
EPA	U.S. Environmental Protection Agency
HPLC	High Performance Liquid Chomatography
ID	Identification
IOP	Internal Operating Procedure
MOR	Modulus of Rupture
Na_2CO_3	Sodium Carbonate
NHSRC	National Homeland Security Research Center
QA	Quality Assurance
QAPP	Quality Assurance Project Plan (QAPP)
QMP	Quality Management Plan
RH	Relative Humidity
SOP	Standing Operating Procedure
UL	Underwriters Laboratories
VHP	Vaporized Hydrogen Peroxide

COUPON SPECIFIC CODING

"W"	bare wood
"R"	carpet
"T"	ceiling suspension tile
"G"	latex-painted gypsum wallboard
"S"	painted structural A572 steel
"C"	unpainted concrete cinder block
"A"	aluminum coupons
"D"	copper coupons
"F"	steel coupons

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APPENDIX A

COUPON IDENTIFIER CODE

All coupons were marked with an ID number that will consist of a nine character alphanumeric code. A description of the identifier pattern and an example code are shown below.

Code Pattern

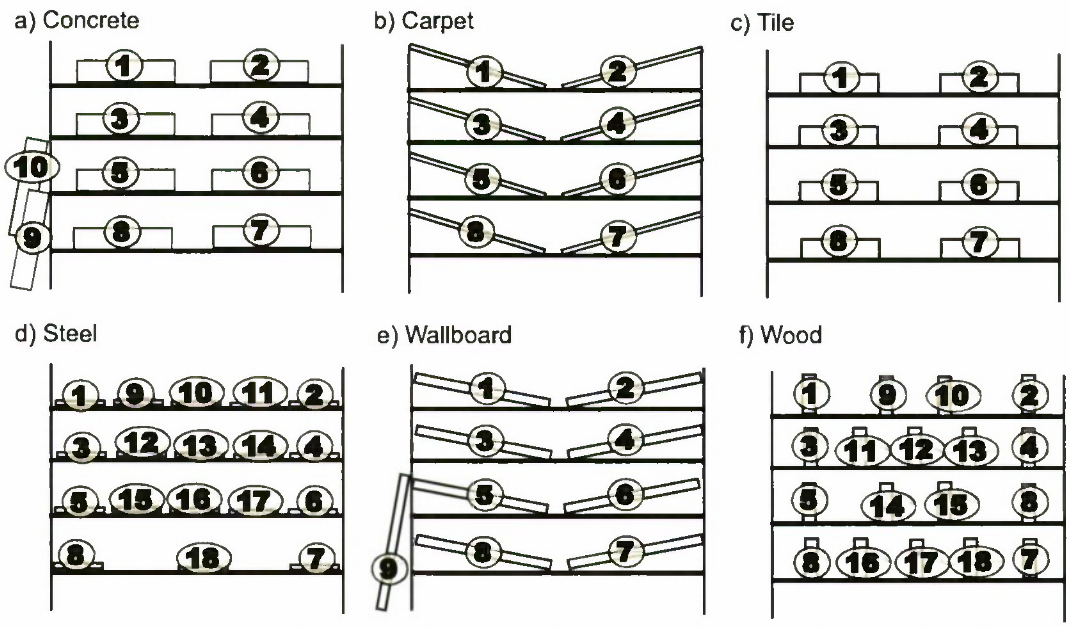
Character	*Explanation*		
1	Material		
	W	=	wood
	G	=	gypsum
	S	=	A572 steel
	T	=	acoustic ceiling tile
	C	=	concrete cinder block
	R	=	carpet
	B	=	circuit breakers
	A	=	Aluminum coupons
	F	=	Copper coupons
	E	=	Steel coupons
2	Fumigant		
	V	=	VHP
	D	=	chlorine dioxide
	N	=	no fumigant
	Test start date		
3	year		for example: 4 = 2004
4,5	month		for example: 06 = June
6,7	day		for example: 10 = the 10^{th} of a month
8,9	Chamber position (see IOP DS04016 figure 1)		

Example GV4101104

Gypsum Wallboard with test start date of 11 Octobcr 2004 and is sample numbcr 4

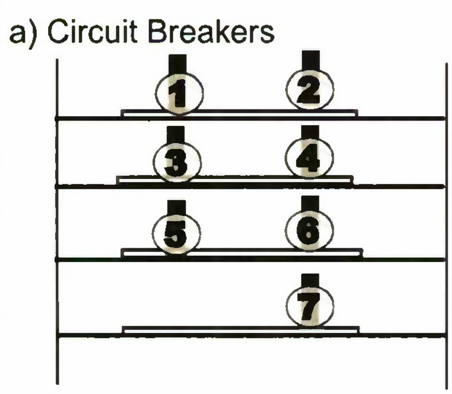
The coupon placement figure taken from the test plan is provided below.

Figure A.1: IOP DS04016 Figure 1, "Coupon Placement in Chambers"



Coupons shown on rack shelves from direction of glove box transfer chamber. Pictoral coupon scaling for length and width is (0.75 * 2 *(cm / 10)).

Figure A.2: IOP DS04016 Fig., "Circuit Breaker Placement in Chambers"



Coupons shown on rack shelves from direction of glove box transfer chamber.

APPENDIX B

DETAILED COUPON PREPARATION AND INSPECTION PROCEDURES

COUPON PREPARATION PROCEDURE: The coupon preparation, unless otherwise noted, was conducted at the U.S. Army Edgewood Chemical Biological Center Experimental Fabrication Shop.

Mechanically Graded Lumber (Bare Wood)

- Stock Item Description: 2 x 4 x 8 KD WW/SPF Stud
- Supplier/Source: Home Depot, Edgewood, MD
- Coupon Dimensions: 10 in. x 1 ½ in. x ½ in.
- Preparation of Coupon: The machined ends of the stock were discarded by removing greater than ¼ in. of the machined end. Coupons were cut from stock using a table saw equipped with an 80 tooth crosscut blade.

Latex-Painted Gypsum Wallboard

- Stock Item Description: ½ in. 4 ft. x 8 ft. Drywall
- Supplier/Source: Home Depot, Edgewood, MD
- Coupon Dimensions: 6 in. x 6 in. x ½ in.
- Preparation of Coupon:
  - The ASTM method requires that the samples be taken from the interior of material rather than from the edge (machined edge). The machined ends of the stock were discarded by cutting away greater than 4 in. from each side.
  - Coupons were cut from stock using a table saw equipped with an 80 tooth crosscut blade.
  - The 6 in. x 6 in. coupons were painted with 1 mil of Glidden PVA Primer and followed by 1–2-mils of Glidden latex topcoat. The primed coupons were allowed to stand for greater than 24 h prior to the application of the topcoat.
  - All six sides of the 6 in. x 6 in. coupon were painted.

Concrete Cinder Block

- Stock Item Description: 8 in. x 16 in. x 1.5 in. concrete cinder block cap
- Supplier/Source: York Supply, Aberdeen MD
- Original Coupon Dimensions: 4 in. x 8 in. x 1.5 in.
- Modified Coupon Dimensions: 4 in. x 8 in. x 0.5 in.
- Preparation of Coupon:
  - Coupons were cut from stock using a water-jet.
  - Four coupons were cut from each stock piece.
  - Original dimensions too large for material testing
    - Each coupon cut into three sections
    - Two sections measured at modified coupon dimensions
    - Third section discarded.

Carpet

- Stock Item Description: 12 ft. Powerhouse 20 Tradewind
- Supplier/Source: Home Depot, Edgewood, MD
- Coupon Dimensions: 6 in. x 8 in.
- Preparation of Coupon:
  - Coupons were cut from the stock using a utility knife.

- The longer direction (8 in.) were cut parallel to the machine edge.
- The machined edge were discarded by removing greater than ½ in.

Painted Structural Steel

- Stock Item Description: A572 Grade 50, 4 ft. x 8 ft. x ¼ in.
- Supplier/Source: Specialized Metals
- Coupon Dimensions: 1/4 in. x 12 in. total, dog bone shaped with 2 in. wide at ends, 0.75 in. width at center
- Preparation of Coupon:
  - Coupons were cut from stock using a water-jet.
  - A visual observation was conducted on each coupon to determine if size and shape deviated from dimension and the coupons were discarded if deviations were evident.
  - Coupons were cleaned and degreased following procedures outlined in TTC-490.
  - Coupons were prepared for painting per TT-P-645 with red oxide primer.

The Edgewood Chemical Biological Center Experimental Fabrication Shop prepared the materials in accordance with the standards used for the preparation and painting of steel. TTC-490 is a Federal Standard providing cleaning methods and pretreatment of iron surfaces for application of organic coatings. The pretreatment is the application of a zinc phosphate corrosion inhibitor. TT-P-645 is a Federal Standard for the application of alkyd paint. These standards were not obtained though this program but were purchased by the Experimental Fabrication Shop for their work.

Ceiling Suspension Tile

- Stock Item Description: Armstrong 954, Classic Fine Textured, 24 in. x 24 in. x 9/16 in.
- Supplier/Source: Home Depot, Edgewood, MD
- Coupon Dimensions: 12 in. x 3 in. x 9/16 in.
- Preparation of Coupon: Coupons were cut from stock using a table saw equipped with an 80 tooth crosscut blade. Sixteen samples were removed from each stock item.

COUPON INSPECTION PROCEDURE: All coupons were inspected prior to testing to ensure that the material being used was in suitable condition. Coupons were rejected if there were cracks, breaks, dents, or defects beyond those typical for the type of material. In addition, coupons were measured to verify the coupon dimensions. Coupons deviating from the following dimension ranges were discarded.

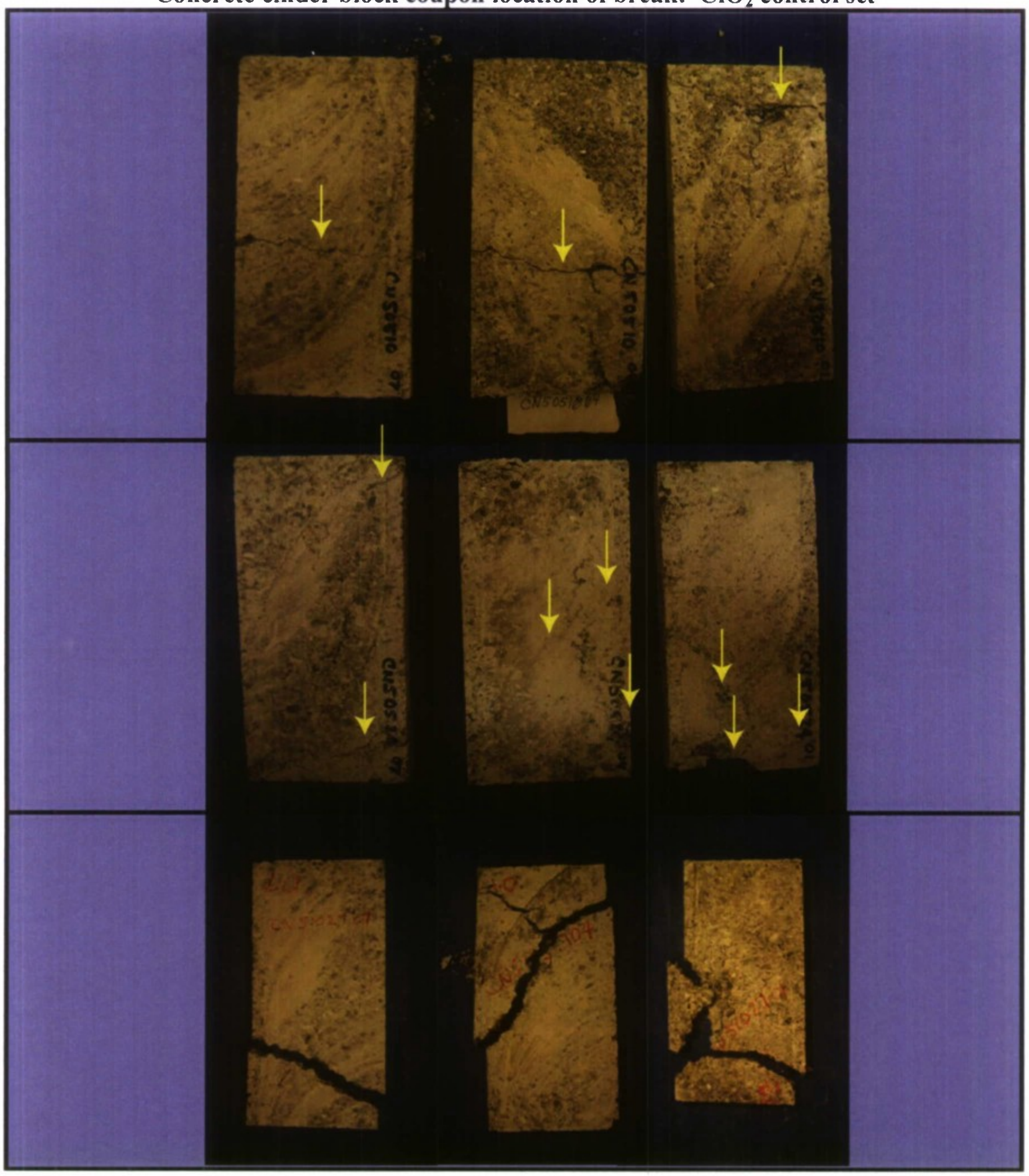
Mechanically Graded Lumber	10 in. ± 1/16 in. x 1.5 in. ± 1/16 in. x 0.5 in. ± 1/32 in.
Latex-Painted Gypsum Wallboard	6 in. ± 1/16 in. x 6 in. ± 1/16 in. x 0.5 in. ± 1/16 in.
Concrete Cinder Block	4 in. ± ½ in. x 8 in. ± ½ in. x 0.5 in. ± 1/16 in.
Carpet	6 in. ± 1/8 in. x 8 in. ± 1/8 in.
Painted Structural Steel	1/4 in. ± 1/128 in. x 12 in. ± 1/16 in. with 2 in. ± 1/16 in. wide at ends, ¾ in. ± 1/16 in. wide in. center
Ceiling Suspension Tile	12 in. ± 1/8 in. x 3 in. ± 1/16 in. x 9/16 in. ± 1/16 in.

APPENDIX C

CONCRETE CINDER BLOCK COUPON BREAK LOCATION

There is no requirement for reporting the loeation of the break; however, eonerete block is a variable material and differences in location were observed. This appendix provides additional information though test photographs. Yellow arrows are used on samples where the photograph contract may not elearly show the loeation of the break.

Concrete cinder block coupon location of break: ClO_2 control set



Concrete cinder block coupon location of break: ClO_2 control set

Concrete cinder block coupon location of break: ClO_2 control set

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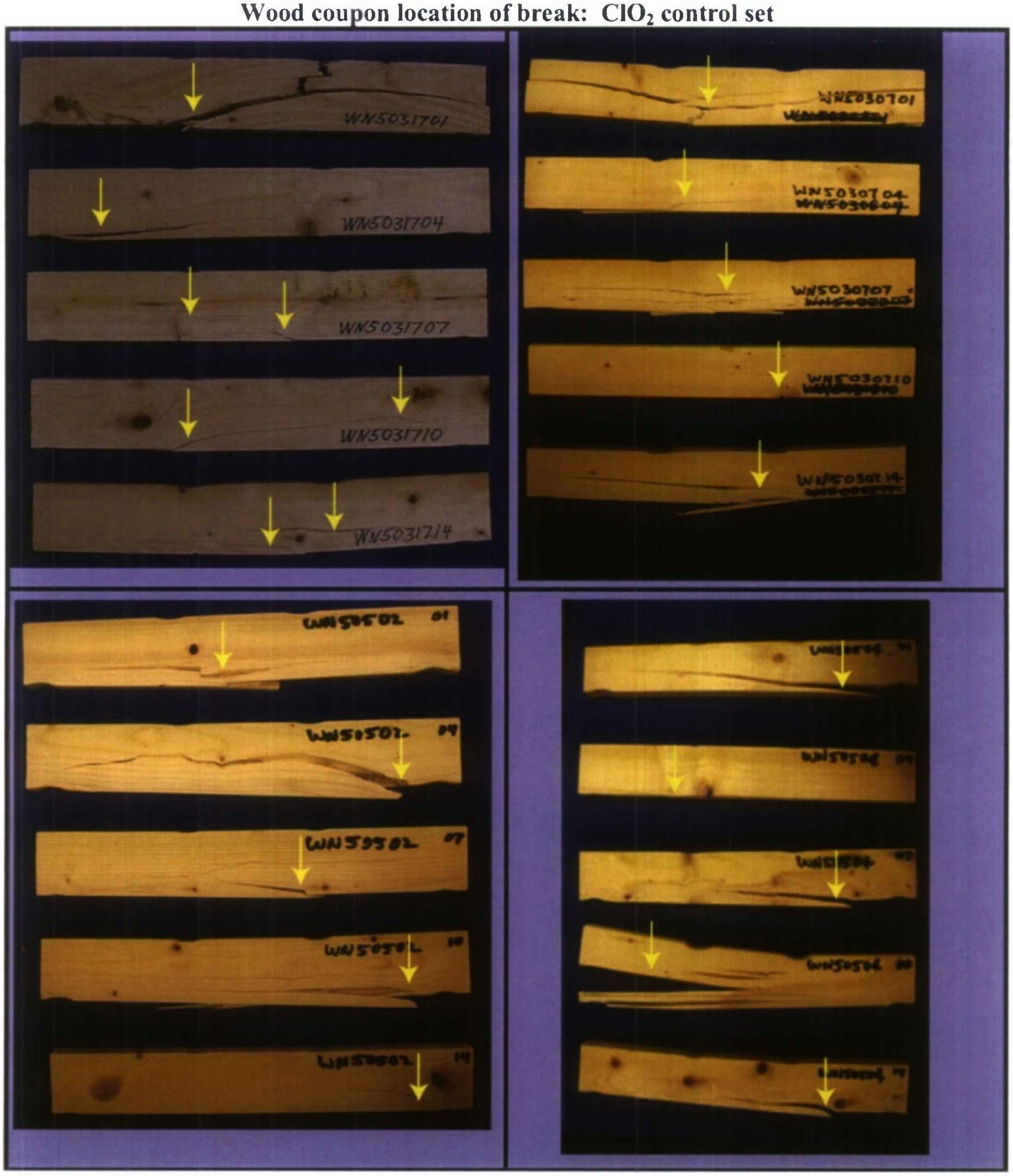
APPENDIX D

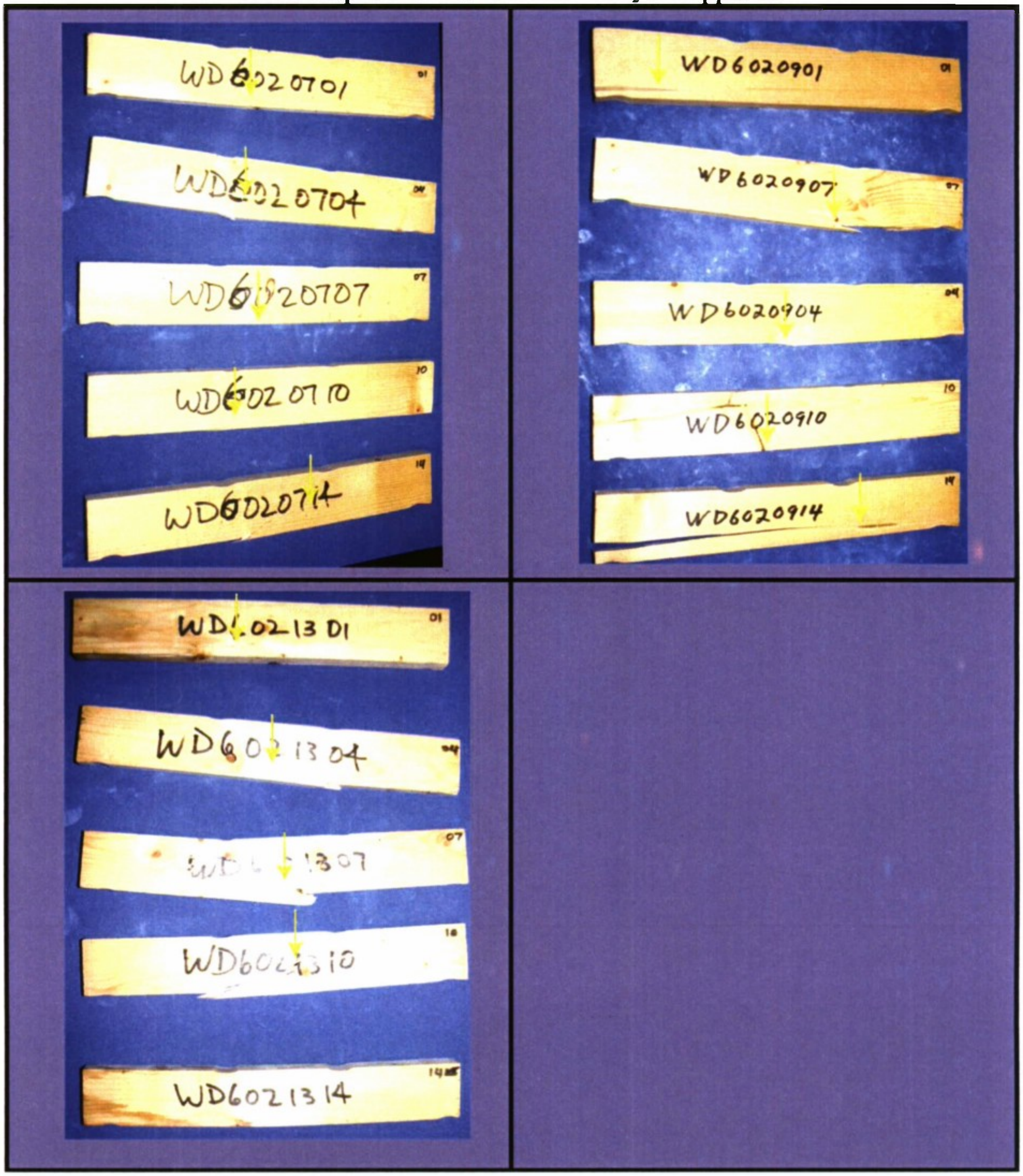
WOOD COUPON LOCATION OF BREAK

The ASTM test method requires reporting the location of the break for each wood sample. This appendix provides this reporting information in pictorial form. Yellow arrows are used on samples where the photograph contract may not clearly show the location of the break.

Wood coupon location of break: ClO_2 control set



Wood coupon location of break: ClO_2 1000 ppm set



Wood coupon location of break: ClO_2 2000 ppm set